

References

Other methods

Contents of Advanced Techniques for Biopolymer Structure and Dynamics

X-ray Diffraction/Scattering

crystallographic

Crystallography is the experimental science of determining the arrangement of atoms in solids. In older usage, it is the scientific study of crystals. The word "crystallography" is derived from the Greek words *crystallon* = cold drop / frozen drop, with its meaning extending to all solids with some degree of transparency, and *graphein* = write.

Before the development of X-ray diffraction crystallography (see below), the study of crystals was based on the geometry of the crystals. This involves measuring the angles of crystal faces relative to theoretical reference axes (crystallographic axes), and establishing the symmetry of the crystal in question. The former is carried out using a goniometer. The position in 3D space of each crystal face is plotted on a stereographic net, e.g. Wulff net or Lambert net. In fact, the pole to each face is plotted on the net. Each point is labelled with its Miller index. The final plot allows the symmetry of the crystal to be established.

Crystallographic methods now depend on the analysis of the diffraction patterns that emerge from a sample that is targeted by a beam of some type. The beam is not always electromagnetic radiation, even though X-rays are the most common choice. For some purposes electrons or neutrons are used, which is possible due to the wave properties of the particles. Crystallographers often explicitly state the type of illumination used when referring to a method, as with the terms **X-ray diffraction**, **neutron diffraction** and **electron diffraction**.

These three types of radiation interact with the specimen in different ways. X-rays interact with the spatial distribution of the valence electrons, while electrons are charged particles and therefore feel the total charge distribution of both the atomic nuclei and the surrounding electrons. Neutrons are scattered by the atomic nuclei through the strong nuclear forces, but in addition, the magnetic moment of neutrons is non-zero. They are therefore also scattered by magnetic fields. When neutrons are scattered from hydrogen-containing materials, they produce diffraction patterns with high noise levels. However, the material can sometimes be treated to substitute hydrogen for deuterium. Because of these different forms of interaction, the three types of radiation are suitable for different crystallographic studies.

Theory

An image of a small object is usually generated by using a lens to focus the illuminating radiation, as is done with the rays of the visible spectrum in light microscopy. However, the wavelength of visible light (about 4000 to 7000 Angstroms) is three orders of magnitude longer than the length of typical atomic bonds and atoms themselves (about 1 to 2 Angstroms). Therefore, obtaining information about the spatial arrangement of atoms requires the use of radiation with shorter wavelengths, such as X-rays. Employing shorter wavelengths implied abandoning microscopy and true imaging, however, because there exists no material from which a lens capable of focusing this type of radiation can be created. (That said, scientists have had some success focusing X-rays with microscopic

Fresnel zone plates made from gold, and by critical-angle reflection inside long tapered capillaries[1]). Diffracted x-ray beams cannot be focused to produce images, so the sample structure must be reconstructed from the diffraction pattern. Sharp features in the diffraction pattern arise from periodic, repeating structure in the sample, which are often very strong due to coherent reflection of many photons from many regularly spaced instances of similar structure, while non-periodic components of the structure result in diffuse (and usually weak) diffraction features.

Because of their highly ordered and repetitive structure, crystals give diffraction patterns of sharp Bragg reflection spots, and are ideal for analyzing the structure of solids.

Notation

See Miller index for a full treatment of this topic.

- Coordinates in *square brackets* such as **[100]** denote a direction vector (in real space).
- Coordinates in *angle brackets* or *chevrons* such as **<100>** denote a *family* of directions which are related by symmetry operations. In the cubic crystal system for example, **<100>** would mean [100], [010], [001] or the negative of any of those directions.
- Miller indices in *parentheses* such as **(100)** denote a plane of the crystal structure, and regular repetitions of that plane with a particular spacing. In the cubic system, the normal to the (hkl) plane is the direction [hkl], but in lower-symmetry cases, the normal to (hkl) is not parallel to [hkl].
- Indices in *curly brackets* or *braces* such as **{100}** denote a family of planes and their normals which are equivalent in cubic materials due to symmetry operations, much the way angle brackets denote a family of directions. In non-cubic materials, <hkl> is not necessarily perpendicular to {hkl}.

Technique

Some materials studied using crystallography, proteins for example, do not occur naturally as crystals. Typically, such molecules are placed in solution and allowed to crystallize over days, weeks, or months through vapor diffusion. A drop of solution containing the molecule, buffer, and precipitants is sealed in a container with a reservoir containing a hygroscopic solution. Water in the drop diffuses to the reservoir, slowly increasing the concentration and allowing a crystal to form. If the concentration were to rise more quickly, the molecule would simply precipitate out of solution, resulting in disorderly granules rather than an orderly and hence usable crystal.

Once a crystal is obtained, data can be collected using a beam of radiation. Although many universities that engage in crystallographic research have their own X-ray producing equipment, synchrotrons are often used as X-ray sources, because of the purer and more complete patterns such sources can generate. Synchrotron sources also have a much higher intensity of X-ray beams, so data collection takes a fraction of the time normally necessary at weaker sources.

Producing an image from a diffraction pattern requires sophisticated mathematics and often an iterative process of **modelling and refinement**. In this process, the mathematically predicted diffraction patterns of an hypothesized or "model" structure are compared to the actual pattern generated by the crystalline sample. Ideally, researchers make several initial guesses, which through refinement all converge on the same answer.

Models are refined until their predicted patterns match to as great a degree as can be achieved without radical revision of the model. This is a painstaking process, made much easier today by computers.

The mathematical methods for the analysis of diffraction data only apply to *patterns*, which in turn result only when waves diffract from orderly arrays. Hence crystallography applies for the most part only to crystals, or to molecules which can be coaxed to crystallize for the sake of measurement. In spite of this, a certain amount of molecular information can be deduced from the patterns that are generated by fibers and powders, which while not as perfect as a solid crystal, may exhibit a degree of order. This level of order can be sufficient to deduce the structure of simple molecules, or to determine the coarse features of more complicated molecules (the double-helical structure of DNA, for example, was deduced from an X-ray diffraction pattern that had been generated by a fibrous sample).

Crystallography in materials engineering

Crystallography is a tool that is often employed by materials scientists. In single crystals, the effects of the crystalline arrangement of atoms is often easy to see macroscopically, because the natural shapes of crystals reflect the atomic structure. In addition, physical properties are often controlled by crystalline defects. The understanding of crystal structures is an important prerequisite for understanding crystallographic defects. Mostly, materials do not occur in a single crystalline, but poly-crystalline form, such that the powder diffraction method plays a most important role in structural determination.

A number of other physical properties are linked to crystallography. For example, the minerals in clay form small, flat, platelike structures. Clay can be easily deformed because the platelike particles can slip along each other in the plane of the plates, yet remain strongly connected in the direction perpendicular to the plates. Such mechanisms can be studied by crystallographic texture measurements.

In another example, iron transforms from a body-centered cubic (bcc) structure to a face-centered cubic (fcc) structure called austenite when it is heated. The fcc structure is a close-packed structure, and the bcc structure is not, which explains why the volume of the iron decreases when this transformation occurs.

Crystallography is useful in phase identification. When performing any process on a material, it may be desired to find out what compounds and what phases are present in the material. Each phase has a characteristic arrangement of atoms. Techniques like X-ray diffraction can be used to identify which patterns are present in the material, and thus which compounds are present (note: the determination of the "phases" within a material should not be confused with the more general problem of "phase determination," which refers to the phase of waves as they diffract from planes within a crystal, and which is a necessary step in the interpretation of complicated diffraction patterns).

Crystallography covers the enumeration of the symmetry patterns which can be formed by atoms in a crystal and for this reason has a relation to group theory and geometry. See symmetry group.

Biology

X-ray crystallography is the primary method for determining the molecular conformations of biological macromolecules, particularly protein and nucleic acids such as DNA and RNA. In fact, the double-helical structure of DNA was deduced from crystallographic data. The first crystal structure of a macromolecule was solved in 1958 (Kendrew, J.C. et al. (1958) A three-dimensional model of the myoglobin molecule obtained by X-ray analysis (*Nature* 181, 662–666). The Protein Data Bank (PDB) is a freely accessible repository for the structures of proteins and other biological macromolecules. Computer programs like RasMol or Pymol can be used to visualize biological molecular structures.

Electron crystallography has been used to determine some protein structures, most notably membrane proteins and viral capsids.

Scientists of note

- William Henry Bragg
 - William Lawrence Bragg
 - Auguste Bravais
 - Francis Crick
 - Pierre Curie
 - Boris Delone
 - Paul Peter Ewald
 - Rosalind Franklin
 - Georges Friedel
 - René Just Haüy
 - Carl Hermann
 - Dorothy Crowfoot Hodgkin
 - Robert Huber
 - Max von Laue
 - Kathleen Lonsdale
 - Ernest-François Mallard
 - Charles-Victor Mauguin
 - William Hallowes Miller
 - Max Perutz
 - Hugo Rietveld
 - Jean-Baptiste L. Romé de l'Isle
 - Constance Tipper
 - Don Craig Wiley
 - Ada Yonath
-

See also

- Atomic packing factor
 - Condensed Matter Physics
 - Crystal engineering
 - Crystal growth
 - Crystal optics
 - Crystal system
 - Crystal
 - Crystallite
 - Crystallization processes
 - Crystallographic database
 - Crystallographic group
 - Diffraction
 - Dynamical theory of diffraction
 - Electron crystallography
 - Electron diffraction
 - Euclidean plane isometry
 - Fixed points of isometry groups in Euclidean space
 - Group action
 - Laser-heated pedestal growth
 - Materials Science
 - Metallurgy
 - Mineralogy
 - Neutron crystallography
 - Neutron diffraction
 - Neutron Diffraction at OPAL
 - Permutation group
 - Point group
 - Powder diffraction
 - Solid state chemistry
 - Space group
 - Symmetric group
 - Symmetry group
 - Symmetry
 - X-ray crystallography
 - X-ray diffraction
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External links

- Introduction to Crystallography and Mineral Crystal Systems ^[2]
 - Crystallographic Teaching Pamphlets ^[3]
 - Crystal Lattice Structures ^[4]
 - Freely Available Crystallographic Software for Academia ^[5]
 - NetSci Software Listing for Crystallography ^[6]
 - SINCRIS Information Server for Crystallography ^[7]
 - ORTEP a professional grade viewer for use on a PC which is based on the FORTRAN code which came from Oak Ridge ^[8]
 - Vega Science Trust Interviews on Crystallography ^[9] Freeview video interviews with Max Pertuz, Rober Huber and Aaron Klug.
 - Commission on Crystallographic Teaching, Pamphlets ^[10]
 - Crystallography site of Steffen Weber with lots of Java Applets ^[11]
 - IUCr Online Dictionary of Crystallography ^[12]
 - American Crystallographic Association ^[13]
 - Laue Measurement of Single-Crystal Turbine Blades ^[14]
 - Ames Laboratory, US DOE Crystallography Research Resources ^[15]
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- [2] <http://www.rockhounds.com/rockshop/xtal/index.html>
- [3] <http://www.mineralogie.uni-wuerzburg.de/links/teach/crysteach.html>
- [4] <http://cst-www.nrl.navy.mil/lattice/spcgrp/>
- [5] <http://www.ccp14.ac.uk/>
- [6] <http://www.netsci.org/Resources/Software/Struct/xray.html>
- [7] <http://www.iucr.org/sincris-top/>
- [8] <http://www.chem.gla.ac.uk/~louis/software/ortep3/download.html>
- [9] <http://www.vega.org.uk>
- [10] <http://www.iucr.org/iucr-top/comm/cteach/pamphlets.html>
- [11] <http://www.jcrystal.com/steffenweber/>
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- [15] <http://www.mcbmm.ameslab.gov/index.html>

scattered

Scattering is a general physical process where some forms of radiation, such as light, sound, or moving particles, are forced to deviate from a straight trajectory by one or more localized non-uniformities in the medium through which they pass. In conventional use, this also includes deviation of reflected radiation from the angle predicted by the law of reflection. Reflections that undergo scattering are often called *diffuse* reflections and unscattered reflections are called *specular* (mirror-like) reflections. The types of non-uniformities which can cause scattering, sometimes known as *scatterers* or *scattering centers*, are too numerous to list, but a small sample includes particles, bubbles, droplets, density fluctuations in fluids, defects in crystalline solids, surface roughness, cells in organisms, and textile fibers in clothing. The effects of such features on the path of almost any type of propagating wave or moving particle can be described in the framework of scattering theory.

Single and multiple scattering

When radiation is only scattered by one localized scattering center, this is called *single scattering*. It is very common that scattering centers are grouped together, and in those cases the radiation may scatter many times, which is known as *multiple scattering*. The main difference between the effects of single and multiple scattering is that single scattering can usually be treated as a random phenomenon and multiple scattering is usually more deterministic. Because the location of a single scattering center is not usually well known relative to the path of the radiation, the outcome, which tends to depend strongly on the exact incoming trajectory, appears random to an observer. This type of scattering would be exemplified by an electron being fired at an atomic nucleus. In that case, the atom's exact position relative to the path of the electron is unknown and would be immeasurable, so the exact direction of the electron after the collision is unknown, plus the quantum-mechanical nature of this particular interaction also makes the interaction random. Single scattering is therefore often described by probability distributions.

With multiple scattering, the randomness of the interaction tends to be averaged out by the large number of scattering events, so that the final path of the radiation appears to be a

deterministic distribution of intensity. This is exemplified by a light beam passing through thick fog. Multiple scattering is highly analogous to diffusion, and the terms *multiple scattering* and *diffusion* are interchangeable in many contexts. Optical elements designed to produce multiple scattering are thus known as *diffusers*.

Not all single scattering is random, however, as a well-controlled laser beam can be exactly positioned to scatter off a microscopic particle with a deterministic outcome. Such situations are encountered in radar scattering as well, where the targets tend to be macroscopic objects such as people or aircraft.

Similarly, multiple scattering can sometimes have somewhat random outcomes, particularly with coherent radiation. The random fluctuations in the multiply-scattered intensity of coherent radiation are called *speckles*. Speckle also occurs if multiple parts of a coherent wave scatter from different centers. In certain rare circumstances, multiple scattering may only involve small number of interactions such that the randomness is not completely averaged out. These systems are considered to be some of the most difficult to model accurately.

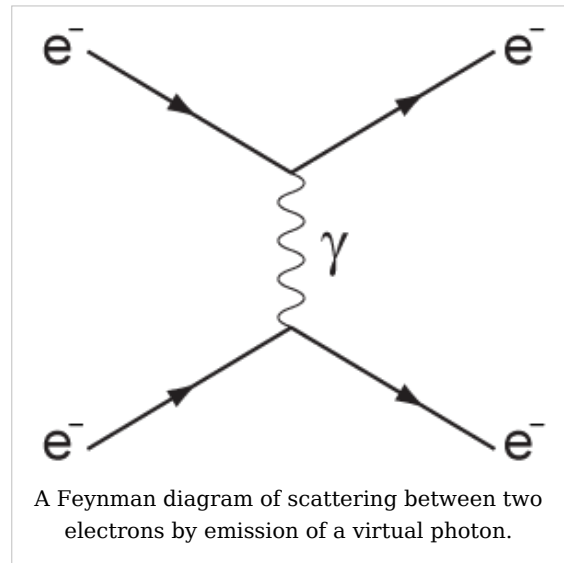
The description of scattering and the distinction between single and multiple scattering are often highly involved with wave-particle duality.

Major research problems in scattering often involve predicting how various systems will scatter radiation, which can almost always be solved given sufficient computing power and knowledge of the system. A widely studied but more difficult challenge is the inverse scattering problem, in which the goal is to observe scattered radiation and use that observation to determine properties of either the scatterer or the radiation before scattering. In general, the inverse is not unique; several different types of scattering centers can usually give rise to the same pattern of scattered radiation, so the problem is not solvable in the general case. Fortunately, there are ways to extract some useful, albeit incomplete, information about the scatterer, and these techniques are widely used for sensing and metrology applications (Colton & Kress 1998).

Some areas where scattering and scattering theory are significant include radar sensing, medical ultrasound, semiconductor wafer inspection, polymerization process monitoring, acoustic tiling, free-space communications, and computer-generated imagery.

Electromagnetic scattering

Electromagnetic (EM) waves are one of the best known and most commonly encountered forms of radiation that undergo scattering. Scattering of light and radio waves (especially in radar) is particularly important. Several different aspects of electromagnetic scattering are distinct enough to have conventional names. Major forms of elastic light scattering (involving negligible energy transfer) are Rayleigh scattering and Mie scattering. Inelastic EM scattering effects include Brillouin scattering, Raman scattering, inelastic X-ray scattering and Compton scattering.



Light scattering is one of the two major physical processes that contribute to the visible appearance of most objects, the other being absorption. Surfaces described as *white* owe their appearance almost completely to the scattering of light by the surface of the object. The absence of surface scattering leads to a shiny or glossy appearance. Light scattering can also give color to some objects, usually shades of blue (as with the sky, the human iris, and the feathers of some birds (Prum et al. 1998)), but resonant light scattering in nanoparticles can produce different highly saturated and vibrant hues, especially when surface plasmon resonance is involved (Roqué et al. 2006).

Rayleigh scattering is a process in which electromagnetic radiation (including light) is scattered by a small spherical volume of variant refractive index, such as a particle, bubble, droplet, or even a density fluctuation. This effect was first modeled successfully by Lord Rayleigh, from whom it gets its name. In order for Rayleigh's model to apply, the sphere must be much smaller in diameter than the wavelength (λ) of the scattered wave; typically the upper limit is taken to be about $1/10$ the wavelength. In this size regime, the exact shape of the scattering center is usually not very significant and can often be treated as a sphere of equivalent volume. The inherent scattering that radiation undergoes passing through a pure gas is due to microscopic density fluctuations as the gas molecules move around, which are normally small enough in scale for Rayleigh's model to apply. This scattering mechanism is the primary cause of the blue color of the Earth's sky on a clear day, as the shorter blue wavelengths of sunlight passing overhead are more strongly scattered than the longer red wavelengths according to Rayleigh's famous $1/\lambda^4$ relation. Along with absorption, such scattering is a major cause of the attenuation of radiation by the atmosphere. The degree of scattering varies as a function of the ratio of the particle diameter to the wavelength of the radiation, along with many other factors including polarization, angle, and coherence.

For larger diameters, the problem of electromagnetic scattering by spheres was first solved by Gustav Mie, and scattering by spheres larger than the Rayleigh range is therefore usually known as Mie scattering. In the Mie regime, the shape of the scattering center becomes much more significant and the theory only applies well to spheres and, with some modification, spheroids and ellipsoids. Closed-form solutions for scattering by certain other

simple shapes exist, but no general closed-form solution is known for arbitrary shapes.

Both Mie and Rayleigh scattering are considered elastic scattering processes, in which the energy (and thus wavelength and frequency) of the light is not substantially changed. However, electromagnetic radiation scattered by moving scattering centers does undergo a Doppler shift, which can be detected and used to measure the velocity of the scattering center/s in forms of techniques such as LIDAR and radar. This shift involves a slight change in energy.

At values of the ratio of particle diameter to wavelength more than about 10, the laws of geometric optics are mostly sufficient to describe the interaction of light with the particle, and at this point the interaction is not usually described as scattering.

For modeling of scattering in cases where the Rayleigh and Mie models do not apply such as irregularly shaped particles, there are many numerical methods that can be used. The most common are finite-element methods which solve Maxwell's equations to find the distribution of the scattered electromagnetic field. Sophisticated software packages exist which allow the user to specify the refractive index or indices of the scattering feature in space, creating a 2- or sometimes 3-dimensional model of the structure. For relatively large and complex structures, these models usually require substantial execution times on a computer.

Another special type of EM scattering is coherent backscattering. This is a relatively obscure phenomenon that occurs when coherent radiation (such as a laser beam) propagates through a medium which has a large number of scattering centers, so that the waves are scattered many times while traveling through it. A thick cloud is a typical example of this sort of multiple-scattering medium. The effect produces a very large peak in the scattering intensity in the direction from the which the wave travels—effectively, the light scatters preferentially back the way it came. For incoherent radiation, the scattering typically reaches a local maximum in the backward direction, but the coherent backscatter peak is two times higher than the level would have been if the light were incoherent. It is very difficult to detect and measure for two reasons. The first is fairly obvious, that it is difficult to measure the direct backscatter without blocking the beam, but there are methods for overcoming this problem. The second is that the peak is usually extremely sharp around the backward direction, so that a very high level of angular resolution is needed for the detector to see the peak without averaging its intensity out over the surrounding angles where the intensity can undergo large dips. At angles other than the backscatter direction, the light intensity is subject to numerous essentially random fluctuations called speckles.

This is one of the most robust interference phenomena that survives multiple scattering, and it is regarded as an aspect of a quantum mechanical phenomenon known as weak localization (Akkermans et al. 1986). In weak localization, interference of the direct and reverse paths leads to a net reduction of light transport in the forward direction. This phenomenon is typical of any coherent wave which is multiple scattered. It is typically discussed for light waves, for which it is similar to the weak localization phenomenon for electrons in disordered (semi)conductors and often seen as the precursor to Anderson (or strong) localization of light. Weak localization of light can be detected since it is manifested as an enhancement of light intensity in the backscattering direction. This substantial enhancement is called the cone of coherent backscattering .

Coherent backscattering has its origin in the interference between direct and reverse paths in the backscattering direction. When a multiply scattering medium is illuminated by a laser beam, the scattered intensity results from the interference between the amplitudes associated with the various scattering paths; for a disordered medium, the interference terms are washed out when averaged over many sample configurations, except in a narrow angular range around exact backscattering where the average intensity is enhanced. This phenomenon, is the result of many sinusoidal two-waves interference patterns which add up. The cone is the Fourier transform of the spatial distribution of the intensity of the scattered light on the sample surface, when the latter is illuminated by a point-like source. The enhanced backscattering relies on the constructive interference between reverse paths. One can make an analogy with a Young's interference experiment, where two diffracting slits would be positioned in place of the "input" and "output" scatterers.

See also

- Bragg diffraction
- Brillouin scattering
- Compton scattering
- CREIL (Coherent Raman Effect on Incoherent Light)
- Dynamic Light Scattering
- Kikuchi line
- Light scattering by particles
- Mie theory
- Neutron scattering
- Powder diffraction
- Raman scattering
- Rayleigh scattering
- Rutherford scattering
- Small-angle scattering
- Tyndall effect
- Thomson scattering
- Wolf effect
- X-ray crystallography

External links

- Research group on light scattering and diffusion in complex systems ^[1]
- Multiple light scattering from a photonic science point of view ^[2]
- Neutron Scattering Web ^[3]

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X-ray

X-radiation (composed of **X-rays**) is a form of electromagnetic radiation. X-rays have a wavelength in the range of 10 to 0.01 nanometers, corresponding to frequencies in the range 30 petahertz to 30 exahertz (3×10^{16} Hz to 3×10^{19} Hz) and energies in the range 120 eV to 120 keV. They are shorter in wavelength than UV rays. In many languages, X-radiation is called **Röntgen radiation** after Wilhelm Conrad Röntgen, who is generally credited as their discoverer, and who had called them X-rays to signify an unknown type of radiation.^{[3] :1-2}

X-rays are primarily used for diagnostic radiography and crystallography. As a result, the term *X-ray* is metonymically used to refer to a radiographic image produced using this method, in addition to the method itself. X-rays are a form of ionizing radiation and as such can be dangerous.

X-rays span 3 decades in wavelength, frequency and energy. From about 0.12 to 12 keV they are classified as soft X-rays, and from about 12 to 120 keV as hard X-rays, due to their penetrating abilities.

The distinction between X-rays and gamma rays has changed in recent decades. Originally, the



Hand mit Ringen (Hand with Rings): print of Wilhelm Röntgen's first "medical" X-ray, of his wife's hand, taken on 22 December 1895 and presented to Professor Ludwig Zehnder of the Physik Institut, University of Freiburg, on 1 January 1896.^{[1] [2]}

electromagnetic radiation emitted by X-ray tubes had a longer wavelength than the

radiation emitted by radioactive nuclei (gamma rays).^[4] So older literature distinguished between X- and gamma radiation on the basis of wavelength, with radiation shorter than some arbitrary wavelength, such as 10^{-11} m, defined as gamma rays.^[5] However, as shorter wavelength continuous spectrum "X-ray" sources such as linear accelerators and longer wavelength "gamma ray" emitters were discovered, the wavelength bands largely overlapped. The two types of radiation are now usually defined by their origin: X-rays are emitted by electrons outside the nucleus, while gamma rays are emitted by the nucleus.^[4] [6] [7] [8]

Units of measure and exposure

The measure of X-rays ionizing ability is called the exposure:

- The coulomb per kilogram (C/kg) is the SI unit of ionizing radiation exposure, and measures the amount of radiation required to create 1 coulomb of charge of each polarity in 1 kilogram of matter.
- The röntgen (R) is an obsolete older traditional unit of exposure, which represented the amount of radiation required to create 1 esu of charge of each polarity in 1 cubic centimeter of dry air. $1 \text{ röntgen} = 2.58 \times 10^{-4} \text{ C/kg}$

However, the effect of ionizing radiation on matter (especially living tissue) is more closely related to the amount of energy deposited rather than the charge. This is called the absorbed dose:

- The gray (Gy) which has units of (J/kg), is the SI unit of absorbed dose which is the amount of radiation required to deposit 1 joule of energy in 1 kilogram of any kind of matter.
- The rad is the (obsolete) corresponding traditional unit, equal to 0.01 J deposited per kg. $100 \text{ rad} = 1 \text{ Gy}$.

The equivalent dose is the measure of the biological effect of radiation on human tissue. For X-rays it is equal to the absorbed dose.

- The sievert (Sv) is the SI unit of equivalent dose, which for X-rays is equal to the gray (Gy).
- The rem is the traditional unit of equivalent dose. For X-rays it is equal to the rad or 0.01 J of energy deposited per kg. $1 \text{ sievert} = 100 \text{ rem}$.

Medical X-rays are a major source of *manmade* radiation exposure, accounting for 58% in the USA in 1987, but since most radiation exposure is natural (82%) it only accounts for 10% of *total* USA radiation exposure.^[9]

Reported dosage due to dental X-rays seems to vary significantly. Depending on the source, a typical dental X-ray of a human results in an exposure of perhaps, 3,^[10] 40,^[11] 300,^[12] or as many as 900^[13] mrem (30 to 9,000 μSv).

Medical physics

X-ray K-series spectral line wavelengths (nm) for some common target materials.

Target	$K\beta_1$	$K\beta_2$	$K\alpha_1$	$K\alpha_2$
Fe	0.17566	0.17442	0.193604	0.193998
Co	0.162079	0.160891	0.178897	0.179285
Ni	0.15001	0.14886	0.165791	0.166175
Cu	0.139222	0.138109	0.154056	0.154439
Zr	0.070173	0.068993	0.078593	0.079015
Mo	0.063229	0.062099	0.070930	0.071359

X-rays are generated by an X-ray tube, a vacuum tube that uses a high voltage to accelerate electrons released by a hot cathode to a high velocity. The high velocity electrons collide with a metal target, the anode, creating the X-rays.^[14] In medical X-ray tubes the target is usually tungsten or a more crack-resistant alloy of rhenium (5%) and tungsten (95%), but sometimes molybdenum for more specialized applications, such as when soft X-rays are needed as in mammography. In crystallography, a copper target is most common, with cobalt often being used when fluorescence from iron content in the sample might otherwise present a problem.

The maximum energy of the produced X-ray photon in keV is limited by the energy of the incident electron, which is equal to the voltage on the tube, so an 80 kV tube can't create higher than 80 keV X-rays. When the electrons hit the target, X-rays are created by two different atomic processes:

1. **X-ray fluorescence:** If the electron has enough energy it can knock an orbital electron out of the inner shell of a metal atom, and as a result electrons from higher energy levels then fill up the vacancy and X-ray photons are emitted. This process produces a discrete spectrum of X-ray frequencies, called spectral lines. The spectral lines generated depends on the target (anode) element used and thus are called characteristic lines. Usually these are transitions from upper shells into K shell (called K lines), into L shell (called L lines) and so on.
2. **Bremsstrahlung:** This is radiation given off by the electrons as they are scattered by the strong electric field near the high-Z (proton number) nuclei. These X-rays have a continuous spectrum. The intensity of the X-rays increases linearly with decreasing frequency, from zero at the energy of the incident electrons, the voltage on the X-ray tube.

So the resulting output of a tube consists of a continuous bremsstrahlung spectrum falling off to zero at the tube voltage, plus several spikes at the characteristic lines. The voltages used in diagnostic X-ray tubes, and thus the highest energies of the X-rays, range from roughly 20 to 150 kV.^[15]

In medical diagnostic applications, the low energy (soft) X-rays are unwanted, since they are totally absorbed by the body, increasing the dose. So a thin metal (often aluminum, but can be one of many X-Ray filters) sheet is placed over the window of the X-ray tube, filtering out the low energy end of the spectrum. This is called *hardening* the beam.

Both X-ray production processes are extremely inefficient ($\sim 1\%$) and thus to produce a usable flux of X-rays plenty of energy has to be wasted into heat, which has to be removed from the X-ray tube.

Radiographs obtained using X-rays can be used to identify a wide spectrum of pathologies. Due to their short wavelength, in medical applications, X-rays act more like a particle than a wave. This is in contrast to their application in crystallography, where their wave-like nature is most important.

To take an X-ray of the bones, short X-ray pulses are shot through a body with radiographic film behind. The bones absorb the most photons by the photoelectric process, because they are more electron-dense. The X-rays that do not get absorbed turn the photographic film from white to black, leaving a white shadow of bones on the film.

To generate an image of the cardiovascular system, including the arteries and veins (angiography) an initial image is taken of the anatomical region of interest. A second image is then taken of the same region after iodinated contrast material has been injected into the blood vessels within this area. These two images are then digitally subtracted, leaving an image of only the iodinated contrast outlining the blood vessels. The radiologist or surgeon then compares the image obtained to normal anatomical images to determine if there is any damage or blockage of the vessel.

A specialized source of X-rays which is becoming widely used in research is synchrotron radiation, which is generated by particle accelerators. Its unique features are brightness many orders of magnitude greater than X-ray tubes, wide spectrum, high collimation, and linear polarization.^[16]

Detectors

Photographic plate

The detection of X-rays is based on various methods. The most commonly known methods are a photographic plate, X-ray film in a cassette, and rare earth screens. Regardless of what is "catching" the image, they are all categorized as "Image Receptors" (IR).

Before computers and before digital imaging, a photographic plate was used to produce radiographic images. The images were produced right on the glass plates. Film replaced these plates and was used in hospitals to produce images. Now computed & digital radiography has started to replace film in medicine, though film technology remains in use in industrial radiography processes (e.g. to inspect welded seams). Photographic plates are a thing of history, and their replacement (intensifying screens) is now becoming part of that same history. Silver (necessary to the radiographic & photographic industry) is a non-renewable resource, that has now been replaced by digital (DR) and computed (CR) technology. Where film required wet processing facilities, these new technologies do not. Archiving of these new technologies also saves space.

Since photographic plates are sensitive to X-rays, they provide a means of recording the image, but require a lot of exposure (to the patient), so intensifying screens were devised. They allow a lower dose to the patient, because the screens take the X-ray information and intensify it so that it can be recorded on film positioned next to the intensifying screen.

The part of the patient to be x-rayed is placed between the X-ray source and the image receptor to produce a shadow of the internal structure of that particular part of the body.

X-rays are partially blocked ("attenuated") by dense tissues such as bone, and pass more easily through soft tissues. Areas where the X-rays strike darken when developed, causing bones to appear lighter than the surrounding soft tissue.

Contrast compounds containing barium or iodine, which are radiopaque, can be ingested in the gastrointestinal tract (barium) or injected in the artery or veins to highlight these vessels. The contrast compounds have high atomic numbered elements in them that (like bone) essentially block the X-rays and hence the once hollow organ or vessel can be more readily seen. In the pursuit of a non-toxic contrast material, many types of high atomic number elements were evaluated. For example, the first time the forefathers used contrast it was chalk, and was used on a cadaver's vessels. Unfortunately, some elements chosen proved to be harmful - for example, thorium was once used as a contrast medium (Thorotrast) - which turned out to be toxic in some cases (causing injury and occasionally death from the effects of thorium poisoning). Modern contrast material has improved, and while there is no way to determine who may have a sensitivity to the contrast, the incidence of "allergic-type reactions" are low. (The risk is comparable to that associated with penicillin.)

Photostimulable phosphors (PSPs)

An increasingly common method of is the use of photostimulated luminescence (PSL), pioneered by Fuji in the 1980s. In modern hospitals a photostimulable phosphor plate (PSP plate) is used in place of the photographic plate. After the plate is x-rayed, excited electrons in the phosphor material remain "trapped" in "colour centres" in the crystal lattice until stimulated by a laser beam passed over the plate surface. The light given off during laser stimulation is collected by a photomultiplier tube and the resulting signal is converted into a digital image by computer technology, which gives this process its common name, computed radiography (also referred to as **digital radiography**). The PSP plate can be reused, and existing X-ray equipment requires no modification to use them.

Geiger counter

Initially, most common detection methods were based on the ionization of gases, as in the Geiger-Müller counter: a sealed volume, usually a cylinder, with a mica, polymer or thin metal window contains a gas, and a wire, and a high voltage is applied between the cylinder (cathode) and the wire (anode). When an X-ray photon enters the cylinder, it ionizes the gas and forms ions and electrons. Electrons accelerate toward the anode, in the process causing further ionization along their trajectory. This process, known as a Townsend avalanche, is detected as a sudden current, called a "count" or "event".

Ultimately, the electrons form a virtual cathode around the anode wire, drastically reducing the electric field in the outer portions of the tube. This halts the collisional ionizations and limits further growth of avalanches. As a result, all "counts" on a Geiger counter are the same size and it can give no indication as to the particle energy of the radiation, unlike the proportional counter. The intensity of the radiation is measurable by the Geiger counter as the counting-rate of the system.

In order to gain energy spectrum information, a diffracting crystal may be used to first separate the different photons. The method is called wavelength dispersive X-ray spectroscopy (WDX or WDS). Position-sensitive detectors are often used in conjunction with dispersive elements. Other detection equipment that is inherently energy-resolving may be

used, such as the aforementioned proportional counters. In either case, use of suitable pulse-processing (MCA) equipment allows digital spectra to be created for later analysis.

For many applications, counters are not sealed but are constantly fed with purified gas, thus reducing problems of contamination or gas aging. These are called "flow counters".

Scintillators

Some materials such as sodium iodide (NaI) can "convert" an X-ray photon to a visible photon; an electronic detector can be built by adding a photomultiplier. These detectors are called "scintillators", filmscreens or "scintillation counters". The main advantage of using these is that an adequate image can be obtained while subjecting the patient to a much lower dose of X-rays.

Image intensification

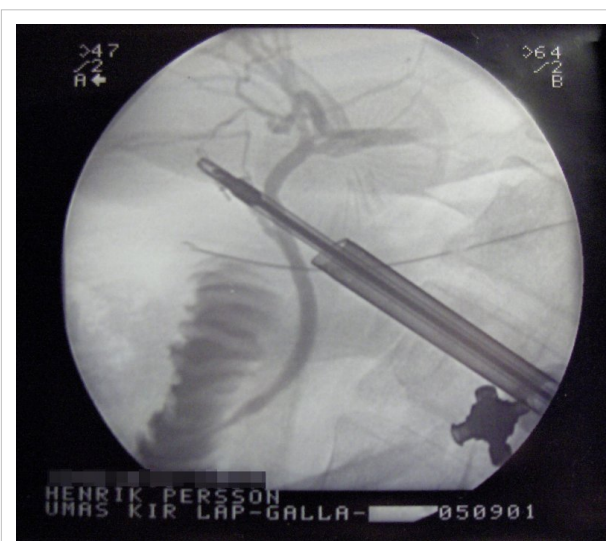
X-rays are also used in "real-time" procedures such as angiography or contrast studies of the hollow organs (e.g. barium enema of the small or large intestine) using fluoroscopy acquired using an X-ray image intensifier. Angioplasty, medical interventions of the arterial system, rely heavily on X-ray-sensitive contrast to identify potentially treatable lesions.

Direct semiconductor detectors

Since the 1970s, new semiconductor detectors have been developed (silicon or germanium doped with lithium, Si(Li) or Ge(Li)). X-ray photons are converted to electron-hole pairs in the semiconductor and are collected to detect the X-rays. When the temperature is low enough (the detector is cooled by Peltier effect or even cooler liquid nitrogen), it is possible to directly determine the X-ray energy spectrum; this method is called energy dispersive X-ray spectroscopy (EDX or EDS); it is often used in small X-ray fluorescence spectrometers. These detectors are sometimes called "solid state detectors". Cadmium telluride (CdTe) and its alloy with zinc, cadmium zinc telluride detectors have an increased sensitivity, which allows lower doses of X-rays to be used.

Practical application in medical imaging didn't start taking place until the 1990s. Currently amorphous selenium is used in commercial large area flat panel X-ray detectors for mammography and chest radiography. Current research and development is focused around pixel detectors, such as CERN's energy resolving Medipix detector.

Note: A standard semiconductor diode, such as a 1N4007, will produce a small amount of current when placed in an X-ray beam. A test device once used by Medical Imaging Service personnel was a small project box that contained several diodes of this type in series, which could be connected to an oscilloscope as a quick diagnostic.



X-ray during Cholecystectomy

Silicon drift detectors (SDDs), produced by conventional semiconductor fabrication, now provide a cost-effective and high resolving power radiation measurement. Unlike conventional X-ray detectors, such as Si(Li)s, they do not need to be cooled with liquid nitrogen.

Scintillator plus semiconductor detectors (indirect detection)

With the advent of large semiconductor array detectors it has become possible to design detector systems using a scintillator screen to convert from X-rays to visible light which is then converted to electrical signals in an array detector. Indirect Flat Panel Detectors (FPDs) are in widespread use today in medical, dental, veterinary and industrial applications.

The array technology is a variant on the amorphous silicon TFT arrays used in many flat panel displays, like the ones in computer laptops. The array consists of a sheet of glass covered with a thin layer of silicon that is in an amorphous or disordered state. At a microscopic scale, the silicon has been imprinted with millions of transistors arranged in a highly ordered array, like the grid on a sheet of graph paper. Each of these thin film transistors (TFTs) are attached to a light-absorbing photodiode making up an individual pixel (picture element). Photons striking the photodiode are converted into two carriers of electrical charge, called electron-hole pairs. Since the number of charge carriers produced will vary with the intensity of incoming light photons, an electrical pattern is created that can be swiftly converted to a voltage and then a digital signal, which is interpreted by a computer to produce a digital image. Although silicon has outstanding electronic properties, it is not a particularly good absorber of X-ray photons. For this reason, X-rays first impinge upon scintillators made from e.g. gadolinium oxysulfide or caesium iodide. The scintillator absorbs the X-rays and converts them into visible light photons that then pass onto the photodiode array.

Visibility to the human eye

While generally considered invisible to the human eye, in special circumstances X-rays can be visible.^[17] Brandes, in an experiment a short time after Röntgen's landmark 1895 paper, reported after dark adaptation and placing his eye close to an X-ray tube, seeing a faint "blue-gray" glow which seemed to originate within the eye itself.^[18] Upon hearing this, Röntgen reviewed his record books and found he too had seen the effect. When placing an X-ray tube on the opposite side of a wooden door Röntgen had noted the same blue glow, seeming to emanate from the eye itself, but thought his observations to be spurious because he only saw the effect when he used one type of tube. Later he realized that the tube which had created the effect was the only one powerful enough to make the glow plainly visible and the experiment was thereafter readily repeatable. The knowledge that X-rays are actually faintly visible to the dark-adapted naked eye has largely been forgotten today; this is probably due to the desire not to repeat what would now be seen as a recklessly dangerous and potentially harmful experiment with ionizing radiation. It is not known what exact mechanism in the eye produces the visibility: it could be due to conventional detection (excitation of rhodopsin molecules in the retina), direct excitation of retinal nerve cells, or secondary detection via, for instance, X-ray induction of phosphorescence in the eyeball with conventional retinal detection of the secondarily produced visible light.

Though X-rays are otherwise invisible it is possible to see the ionization of the air molecules if the intensity of the X-ray beam is high enough. The beamline from the wiggler at the ID11^[19] at ESRF is one example of such high intensity.^[20]

Medical uses

Since Röntgen's discovery that X-rays can identify bony structures, X-rays have been developed for their use in medical imaging. Radiology is a specialized field of medicine. Radiologists employ radiography and other techniques for diagnostic imaging. This is probably the most common use of X-ray technology.

X-rays are especially useful in the detection of pathology of the skeletal system, but are also useful for detecting some disease processes in soft tissue. Some notable examples are the very common chest X-ray, which can be used to identify lung diseases such as pneumonia, lung cancer or pulmonary edema, and the abdominal X-ray, which can detect intestinal obstruction, free air (from visceral perforations) and free fluid (in ascites). X-rays may also be used to detect pathology such as gallstones (which are rarely radiopaque) or kidney stones (which are often visible, but not always). Traditional plain X-rays are less useful in the imaging of soft tissues such as the brain or muscle. Imaging alternatives for soft tissues are computed axial tomography (CAT or CT scanning), magnetic resonance imaging (MRI) or ultrasound. Since 2005, X-rays are listed as a carcinogen by the U.S. government.^[21] The use of X-rays as a treatment is known as radiotherapy and is largely used for the management (including palliation) of cancer; it requires higher radiation energies than for imaging alone.



X-Ray Image of the Paranasal Sinuses, Lateral Projection

X-rays are relatively safe investigation and the radiation exposure is low. But in pregnant patients, the benefits of the investigation (x-ray) should be balanced with the potential hazards to the unborn fetus.^{[22] [23]}

Shielding against X-Rays

Lead is the most common shield against X-Rays because of its high density (11340 kg/m^3), ease of installation and low cost. The maximum range of a high-energy photon such as an X-ray in matter is infinite - at every point in the matter traversed by the photon, there is a probability of interaction. Thus there is a very small probability of no interaction over very large distances. The shielding of photons is therefore exponential - doubling the thickness of shielding will square the shielding effect.

The following table shows the recommended thickness of lead shielding in function of X-Ray energy, from the Recommendations by the Second International Congress of Radiology.^[24]

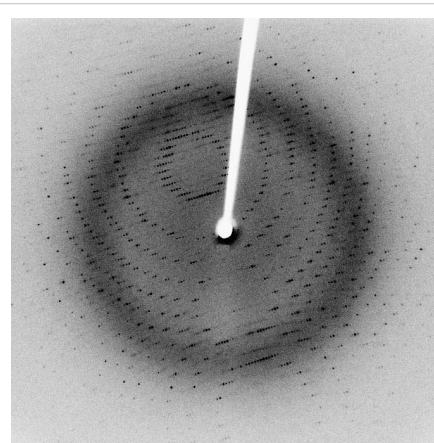
X-Rays generated by peak voltages not exceeding	Minimum thickness of Lead
75 kV	1.0 mm
100 kV	1.5 mm

125 kV	2.0 mm
150 kV	2.5 mm
175 kV	3.0 mm
200 kV	4.0 mm
225 kV	5.0 mm
300 kV	9.0 mm
400 kV	15.0 mm
500 kV	22.0 mm
600 kV	34.0 mm
900 kV	51.0 mm

Other uses

Other notable uses of X-rays include

- X-ray crystallography in which the pattern produced by the diffraction of X-rays through the closely spaced lattice of atoms in a crystal is recorded and then analyzed to reveal the nature of that lattice. A related technique, fiber diffraction, was used by Rosalind Franklin to discover the double helical structure of DNA.^[25]
- X-ray astronomy, which is an observational branch of astronomy, which deals with the study of X-ray emission from celestial objects.
- X-ray microscopic analysis, which uses electromagnetic radiation in the soft X-ray band to produce images of very small objects.
- X-ray fluorescence, a technique in which X-rays are generated within a specimen and detected. The outgoing energy of the X-ray can be used to identify the composition of the sample.
- Industrial radiography uses X-rays for inspection of industrial parts, particularly welds.
- Paintings are often x-rayed to reveal the underdrawing and pentimenti or alterations in the course of painting, or by later restorers. Many pigments such as lead white show well in X-ray photographs.
- Airport security luggage scanners use X-rays for inspecting the interior of luggage for security threats before loading on aircraft.
- X-ray fine art photography
- Roentgen Stereophotogrammetry is used to track movement of bones based on the implantation of markers



Each dot, called a reflection, in this diffraction pattern forms from the constructive interference of scattered X-rays passing through a crystal. The data can be used to determine the crystalline structure.

History

X-rays were discovered emanating from Crookes tubes, experimental discharge tubes invented around 1875, by scientists investigating the cathode rays, that is energetic electron beams, that were first created in the tubes. Crookes tubes created electrons by ionization of the residual air in the tube by a high DC voltage of anywhere between a few kilovolts and 100 kV. This voltage accelerated the electrons coming from the cathode to a high enough velocity that they created X-rays when they struck the anode or the glass wall of the tube. Many of the early Crookes tubes undoubtedly radiated X-rays, because early researchers noticed effects that were attributable to them, as detailed below, before Wilhelm Röntgen first systematically studied them in 1895. Among the important early researchers in X-rays were Ivan Pulyui, William Crookes, Johann Wilhelm Hittorf, Eugen Goldstein, Heinrich Hertz, Philipp Lenard, Hermann von Helmholtz, Nikola Tesla, Thomas Edison, Charles Glover Barkla, Max von Laue, and Wilhelm Conrad Röntgen.



X-ray fine art photography of needlefish by Peter Dazeley

Wilhelm Röntgen

On November 8, 1895, German physics professor Wilhelm Conrad Röntgen, stumbled on X-rays while experimenting with Lenard and Crookes tubes and began studying them. He wrote an initial report "*On a new kind of ray: A preliminary communication*" and on December 28, 1895 submitted it to the Würzburg's Physical-Medical Society journal.^[26] This was the first paper written on X-rays. Röntgen referred to the radiation as "X", to indicate that it was an unknown type of radiation. The name stuck, although (over Röntgen's great objections), many of his colleagues suggested calling them **Röntgen rays**. They are still referred to as such in many languages, including German. Röntgen received the first Nobel Prize in Physics for his discovery.

There are conflicting accounts of his discovery because Röntgen had his lab notes burned after his death, but this is a likely reconstruction by his biographers:^[27] Röntgen was investigating cathode rays with a fluorescent screen painted with barium platinocyanide and a Crookes tube which he had wrapped in black cardboard so the visible light from the tube wouldn't interfere. He noticed a faint green glow from the screen, about 1 meter away. He realized some invisible rays coming from the tube were passing through the cardboard to make the screen glow. He found they could also pass through books and papers on his desk. Röntgen threw himself into investigating these unknown rays systematically. Two months after his initial discovery, he published his paper.

Röntgen discovered its medical use when he saw a picture of his wife's hand on a photographic plate formed due to X-rays. His wife's hand's photograph was the first ever photograph of a human body part using X-rays.

Johann Hittorf

German physicist Johann Hittorf (1824 - 1914), a coinventor and early researcher of the Crookes tube, found when he placed unexposed photographic plates near the tube, that some of them were flawed by shadows, though he did not investigate this effect.

Ivan Pulyui

In 1877 Ukrainian-born Pulyui, a lecturer in experimental physics at the University of Vienna, constructed various designs of vacuum discharge tube to investigate their properties.^[28] He continued his investigations when appointed professor at the Prague Polytechnic and in 1886 he found that that sealed photographic plates became dark when exposed to the emanations from the tubes. Early in 1896, just a few weeks after Röntgen published his first X-ray photograph, Pulyui published high-quality x-ray images in journals in Paris and London.^[28] Although Pulyui had studied with Röntgen at the University of Strasbourg in the years 1873-75, his biographer Gaida (1997) asserts that his subsequent research was conducted independently.^[28]

The first medical X-ray made in the United States was obtained using a discharge tube of Pulyui's design. In January 1896, on reading of Röntgen's discovery, Frank Austin of Dartmouth College tested all of the discharge tubes in the physics laboratory and found that only the Pulyui tube produced X-rays. This was a result of Pulyui's inclusion of an oblique "target" of mica, used for holding samples of fluorescent material, within the tube. On 3 February 1896 Gilman Frost, professor of medicine at the college, and his brother Edwin Frost, professor of physics, exposed the wrist of Eddie McCarthy, whom Edwin had treated some weeks earlier for a fracture, to the X-rays and collected the resulting image of the broken bone on gelatin photographic plates obtained from Howard Langill, a local photographer also interested in Röntgen's work.^[29]

Nikola Tesla

In April 1887, Nikola Tesla began to investigate X-rays using high voltages and tubes of his own design, as well as Crookes tubes. From his technical publications, it is indicated that he invented and developed a special single-electrode X-ray tube,^{[30] [31]} which differed from other X-ray tubes in having no target electrode. The principle behind Tesla's device is called the Bremsstrahlung process, in which a high-energy secondary X-ray emission is produced when charged particles (such as electrons) pass through matter. By 1892, Tesla performed several such experiments, but he did not categorize the emissions as what were later called X-rays. Tesla generalized the phenomenon as radiant energy of "invisible" kinds.^{[32] [33]} Tesla stated the facts of his methods concerning various experiments in his 1897 X-ray lecture before the New York Academy of Sciences.^[34] Also in this lecture, Tesla stated the method of construction and safe operation of X-ray equipment. His X-ray experimentation by vacuum high field emissions also led him to alert the scientific community to the biological hazards associated with X-ray exposure.^[35]

Fernando Sanford

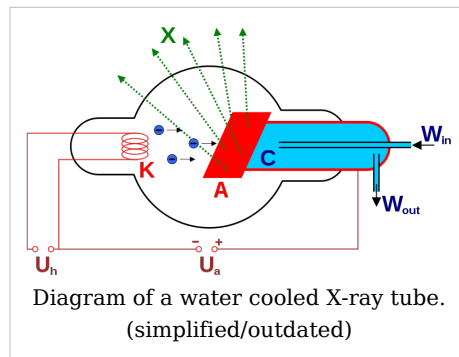
X-rays were generated and detected by Fernando Sanford (1854-1948), the foundation Professor of Physics at Stanford University, in 1891. From 1886 to 1888 he had studied in the Hermann Helmholtz laboratory in Berlin, where he became familiar with the cathode rays generated in vacuum tubes when a voltage was applied across separate electrodes, as previously studied by Heinrich Hertz and Philipp Lenard. His letter of January 6, 1893 (describing his discovery as "electric photography") to *The Physical Review* was duly published and an article entitled *Without Lens or Light, Photographs Taken With Plate and Object in Darkness* appeared in the *San Francisco Examiner*.^[36]

Philipp Lenard

Philipp Lenard, a student of Heinrich Hertz, wanted to see whether cathode rays could pass out of the Crookes tube into the air. He built a Crookes tube (later called a "Lenard tube") with a "window" in the end made of thin aluminum, facing the cathode so the cathode rays would strike it.^[37] He found that something came through, that would expose photographic plates and cause fluorescence. He measured the penetrating power of these rays through various materials. It has been suggested that at least some of these "Lenard rays" were actually X-rays.^[38] Hermann von Helmholtz formulated mathematical equations for X-rays. He postulated a dispersion theory before Röntgen made his discovery and announcement. It was formed on the basis of the electromagnetic theory of light.^[39] However, he did not work with actual X-rays.

Thomas Edison

In 1895, Thomas Edison investigated materials' ability to fluoresce when exposed to X-rays, and found that calcium tungstate was the most effective substance. Around March 1896, the fluoroscope he developed became the standard for medical X-ray examinations. Nevertheless, Edison dropped X-ray research around 1903 after the death of Clarence Madison Dally, one of his glassblowers. Dally had a habit of testing X-ray tubes on his hands, and acquired a cancer in them so tenacious that both arms were amputated in a futile attempt to save his life. "At the 1901 Pan-American Exposition in Buffalo, New York, an assassin shot President William McKinley twice at close range with a .32 caliber revolver." The first bullet was removed but the second remained lodged somewhere in his stomach. McKinley survived for some time and requested that Thomas Edison "rush an X-ray machine to Buffalo to find the stray bullet. It arrived *but wasn't used* . . . McKinley died of septic shock due to bacterial infection."^[40]



The 20th century and beyond

The many applications of X-rays immediately generated enormous interest. Workshops began making specialized versions of Crookes tubes for generating X-rays, and these first generation cold cathode or Crookes X-ray tubes were used until about 1920.

Crookes tubes were unreliable. They had to contain a small quantity of gas (invariably air) as a current will not flow in such a tube if they are fully evacuated. However as time passed the X-rays caused the glass to absorb the gas, causing the tube to generate "harder" X-rays until it soon stopped operating. Larger and more frequently used tubes were provided with devices for restoring the air, known as "softeners". This often took the form of a small side tube which contained a small piece of mica – a substance that traps comparatively large quantities of air within its structure. A small electrical heater heated the mica and caused it to release a small amount of air restoring the tube's efficiency. However the mica itself had a limited life and the restore process was consequently difficult to control.

In 1904, John Ambrose Fleming invented the thermionic diode valve (vacuum tube). This used a hot cathode which permitted current to flow in a vacuum. This idea was quickly applied X-ray tubes, and heated cathode X-ray tubes, called Coolidge tubes, replaced the troublesome cold cathode tubes by about 1920.

Two years later, physicist Charles Barkla discovered that X-rays could be scattered by gases, and that each element had a characteristic X-ray. He won the 1917 Nobel Prize in Physics for this discovery. Max von Laue, Paul Knipping and Walter Friedrich observed for the first time the diffraction of X-rays by crystals in 1912. This discovery, along with the early works of Paul Peter Ewald, William Henry Bragg and William Lawrence Bragg gave birth to the field of X-ray crystallography. The Coolidge tube was invented the following year by William D. Coolidge which permitted continuous production of X-rays; this type of tube is still in use today.



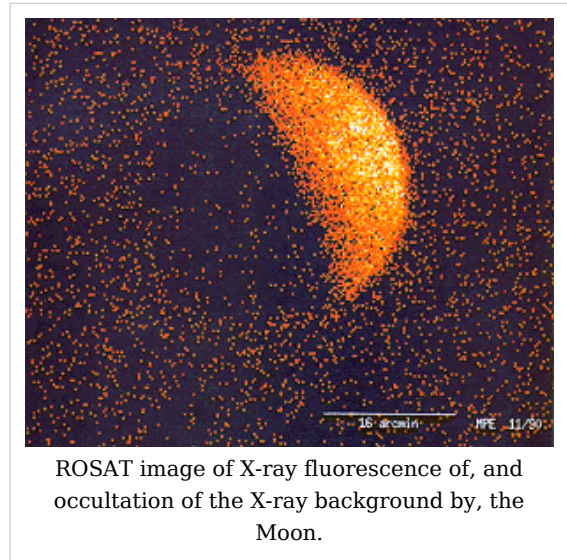
A male technician taking an x-ray of a female patient in 1940. This image was used to argue that exposure to radiation during the x-ray procedure would be a myth.

The use of X-rays for medical purposes (to develop into the field of radiation therapy) was pioneered by Major John Hall-Edwards in Birmingham, England. In 1908, he had to have his left arm amputated owing to the spread of X-ray dermatitis[41].

The X-ray microscope was invented in the 1950s.

The Chandra X-ray Observatory, launched on July 23, 1999, has been allowing the exploration of the very violent processes in the universe which produce X-rays. Unlike visible light, which is a relatively stable view of the universe, the X-ray universe is unstable, it features stars being torn apart by black holes, galactic collisions, and novas, neutron stars that build up layers of plasma that then explode into space.

An X-ray laser device was proposed as part of the Reagan Administration's Strategic Defense Initiative in the 1980s, but the first and only test of the device (a sort of laser "blaster", or death ray, powered by a thermonuclear explosion) gave inconclusive results. For technical and political reasons, the overall project (including the X-ray laser) was de-funded (though was later revived by the second Bush Administration as National Missile Defense using different technologies).



See also

- Neutron radiation
- High energy X-rays
- X-ray crystallography
- X-ray astronomy
- X-ray machine
- X-ray microscope
- X-ray optics
- Backscatter X-ray
- Small angle X-ray scattering (SAXS)
- Geiger counter
- N-ray
- Radiography
- Radiology
- Radiologic technologist
- X-ray vision
- X-ray absorption spectroscopy
- X-Ray filters

Notes

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External links

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- An Example of a Radiograph (http://www.onlinetelemedicine.com/html/product/sam_images/X-Ray.jpg)
- A Photograph of an X-ray Machine (<http://www.iuk.edu/~koalhe/img/Equipment/xray.jpg>)

- An X-ray tube demonstration (Animation) (http://www.ionactive.co.uk/multi-media_video.html?m=4)
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 - Cathode Ray Tube Collection (<http://members.chello.nl/~h.dijkstra19/page5.html>)
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X-ray diffraction techniques

Crystal

A **crystal** or **crystalline solid** is a solid material whose constituent atoms, molecules, or ions are arranged in an orderly repeating pattern extending in all three spatial dimensions. The scientific study of crystals and crystal formation is crystallography. The process of crystal formation via mechanisms of crystal growth is called crystallization or solidification.

The word *crystal* is derived from the ancient Greek word κρύσταλλος (*krustallos*), which had the same meaning, but according to the ancient understanding of crystal. At root it means anything congealed by freezing, such as *ice*.^[1] The word once referred particularly to quartz, or "rock crystal".

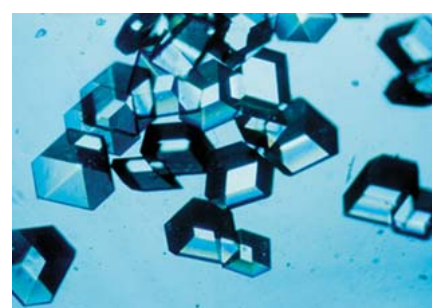
Most metals encountered in everyday life are polycrystals. Crystals are often symmetrically intergrown to form crystal twins.



Quartz crystal. The individual grains of this polycrystalline mineral sample are clearly visible.

Crystal structure

The process of forming a crystalline structure from a fluid or from materials dissolved in the fluid is often referred to as **crystallization**. In the ancient example referenced by the root meaning of the word crystal, water being cooled undergoes a phase change from liquid to solid beginning with small ice crystals that grow until they fuse, forming a polycrystalline structure. The physical properties of the ice depend on the size and arrangement of the individual crystals, or grains, and the same may be said of metals solidifying from a molten state.



Insulin crystals

Which crystal structure the fluid will form depends on the chemistry of the fluid, the conditions under which it is being solidified, and also on the ambient pressure. While the cooling process usually results in the generation of a crystalline material, under certain conditions, the fluid may be frozen in a noncrystalline state. In most cases, this involves cooling the fluid so rapidly that atoms cannot travel to their lattice sites before they lose mobility.

A noncrystalline material,

which has no long-range order, is called an amorphous, vitreous, or glassy material. It is also often referred to as an amorphous solid, although there are distinct differences between solids and glasses: most notably, the process of forming a glass does not release the latent heat of fusion.



Halite (sodium chloride) - a single, large crystal

Crystalline structures occur in all classes of materials, with all types of chemical bonds. Almost all metal exists in a polycrystalline state; amorphous or single-crystal metals must be produced synthetically, often with great difficulty. Ionically bonded crystals can form upon solidification of salts, either from a molten fluid or when it condenses from a solution. Covalently bonded crystals are also very common, notable examples being diamond, silica, and graphite. Polymer materials generally will form crystalline regions, but the lengths of the molecules usually prevent complete crystallization. Weak Van der Waals forces can also play a role in a crystal structure; for example, this type of bonding loosely holds together the hexagonal-patterned sheets in graphite.

Most crystalline materials have a variety of crystallographic defects. The types and structures of these defects can have a profound effect on the properties of the materials.

Crystal phases or forms

See: Phase transformations in solids

Polymorphism is the ability of a solid to exist in more than one crystal form. For example, water ice is ordinarily found in the hexagonal form Ice I_h , but can also exist as the cubic Ice I_c , the rhombohedral ice II, and many other forms.

Amorphous phases are also possible with the same molecule, such as amorphous ice. In this case, the phenomenon is known as polyamorphism.

For pure chemical elements, polymorphism is known as allotropy. For example, diamond, graphite, and fullerenes are different allotropes of carbon.

Other meanings and characteristics

Since the initial discovery of crystal-like individual arrays of atoms that are not regularly repeated, made in 1982 by Dan Shechtman, the acceptance of the concept and the word quasicrystal have led the International Union of Crystallography to redefine the term crystal to mean "any solid having an essentially discrete diffraction diagram", thereby shifting the essential attribute of crystallinity from position space to Fourier space. Within the family of crystals one distinguishes between traditional crystals, which are periodic, or repeating, at the atomic scale, and aperiodic (incommensurate) crystals which are not. This broader definition adopted in 1996 reflects the current understanding that microscopic periodicity is a sufficient but not a necessary condition for crystals.



A large monocrystal of potassium dihydrogen phosphate grown from solution by Saint-Gobain for the megajoule laser of CEA.



Gallium, a metal that easily forms large single crystals



Ice crystals

While the term "crystal" has a precise meaning within materials science and solid-state physics, colloquially "crystal" refers to solid objects that exhibit well-defined and often pleasing geometric shapes. In this sense of the word, many types of crystals are found in nature. The shape of these crystals is dependent on the types of molecular bonds between the atoms to determine the structure, as well as on the conditions under which they formed. Snowflakes, diamonds, and table salt are common examples of crystals.

Some crystalline materials may exhibit special electrical properties such as the ferroelectric effect or the piezoelectric effect. Additionally, light passing through a crystal is often refracted or bent in different directions, producing an array of colors; crystal optics is the study of these effects. In periodic dielectric structures a range of unique optical properties can be expected as seen in photonic crystals.

Crystalline rocks

Inorganic matter, if free to take that physical state in which it is most stable, tends to crystallize. There is no practical limit to the size a crystal may attain under the right conditions, and selenite single crystals in excess of 10 m are found in the Cave of the Crystals in Naica, Mexico.^[2]

Crystalline rock masses have consolidated from aqueous solution or from molten magma. The vast majority of igneous rocks belong to this group and the degree of crystallization depends primarily on the conditions under which they solidified. Such rocks as granite, which have cooled very slowly and under great pressures, have completely crystallized, but many lavas were poured out at the surface and cooled very rapidly; in this latter group a small amount of amorphous or glassy matter is frequent. Other crystalline rocks, the evaporites such as rock salt, gypsum and some limestones have been deposited from aqueous solution, mostly owing to evaporation in arid climates. Still another group, the metamorphic rocks which includes the marbles, mica-schists and quartzites; are recrystallized, that is to say, they were at first fragmental rocks, like limestone, shale and sandstone and have never been in a molten condition nor entirely in solution. The high temperature and pressure conditions of metamorphism have acted on them erasing their original structures, and inducing recrystallization in the solid state.^[1]



Fossil shell with calcite crystals

Properties

Crystal	Particles	Attractive forces	Melting point	Other properties
Ionic	Positive and negative ions	Electrostatic attractions	High	Hard, brittle, good electrical conductor in molten state
Molecular	Polar molecules	London force and dipole-dipole attraction	Low	Soft, non-conductor or extremely poor conductor of electricity in liquid state
Molecular	Non-polar molecules	London force	Low	Soft conductor

See also

- Amorphous solid
- Artificial Snow Crystal
- Atomic packing factor
- Biom mineralisation
- Colloidal crystal
- Crystal growth
- Crystal habit
- Crystal system
- Glass
- Physics of glass
- Inorganic Crystal Structure Database
- Laser Heated Pedestal Growth
- Lead crystal
- Liquid crystal
- Metallic crystal
- Micro-Pulling-Down

- Crystallite
- Crystallographic database
- Quasicrystal
- Seed crystal
- Single crystal

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Dynamical theory of diffraction

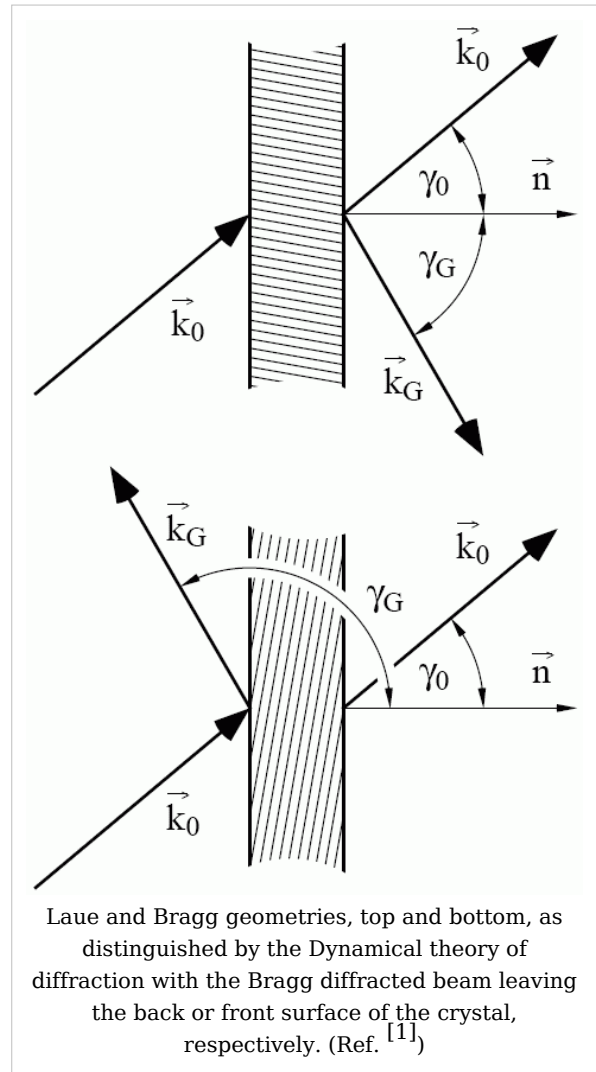
The **dynamical theory of diffraction** describes the interaction of waves with a regular lattice. The wave fields traditionally described are X-rays, neutrons or electrons and the regular lattice atomic crystal structures or nanometer scaled multi-layers or self arranged systems. In a wider sense, similar treatment is related to the interaction of light with optical band-gap materials or related wave problems in acoustics.

Principle of theory

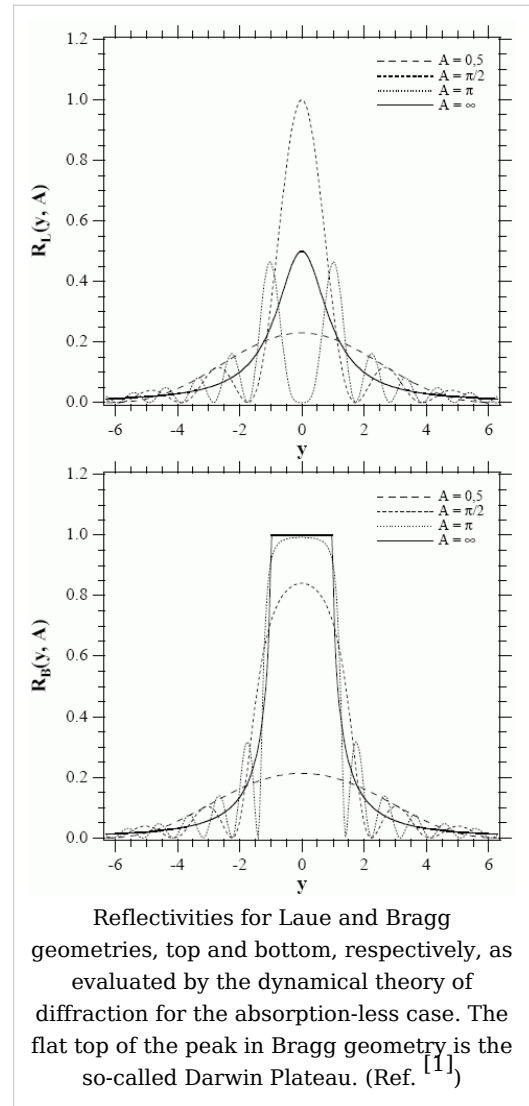
The dynamical theory of diffraction considers the wave field in the periodic potential of the crystal and takes into account all multiple scattering effects. Unlike the kinematic theory of diffraction which describes the approximate position of Bragg or Laue diffraction peaks in reciprocal space, dynamical theory corrects for refraction, shape and width of the peaks, extinction and interference effects. Graphical representations are described in dispersion surfaces around reciprocal lattice points which fulfill the boundary conditions at the crystal interface.

Outcomes

- The crystal potential by itself leads to refraction and specular reflection of the waves at the interface to the crystal and delivers the refractive index off the Bragg reflection. It also corrects for refraction at the Bragg condition and combined Bragg and specular reflection in grazing incidence geometries.
- A Bragg reflection is the splitting of the dispersion surface at the border of the Brillouin zone in reciprocal space. There is a gap between the dispersion surfaces in which no travelling waves are allowed. For a non-absorbing crystal, the reflection curve shows a range of total reflection, the so-called Darwin plateau. Regarding the quantum mechanical energy of the system, this leads to the band gap structure which is commonly well known for electrons.



- Upon Laue diffraction, intensity is shuffled from the forward diffracted beam into the Bragg diffracted beam until extinction. The diffracted beam itself fulfills the Bragg condition and shuffles intensity back into the primary direction. This round-trip period is called the *Pendellösung* period.
- The **extinction length** is related to the *Pendellösung* period. Even if a crystal is infinitely thick, only the crystal volume within the extinction length contributes considerably to the diffraction in Bragg geometry.
- In Laue geometry, beam paths lie within the Borrmann triangle. Kato fringes are the intensity patterns due to *Pendellösung* effects at the exit surface of the crystal.
- Anomalous absorption effects take place due to a standing wave patterns of two wave fields. Absorption is stronger if the standing wave has its anti-nodes on the lattice planes, i.e. where the absorbing atoms are, and weaker, if the anti-nodes are shifted between the planes. The standing wave shifts from one condition to the other on each side of the Darwin plateau which gives the latter an asymmetric shape.



Applications

- X-ray diffraction
- Neutron diffraction
- Electron diffraction and transmission electron microscopy
- Structure determination in Crystallography
- grazing incidence diffraction
- X-ray standing waves
- neutron and X-ray interferometry.
- synchrotron crystal optics
- neutron and X-ray diffraction topography
- X-ray imaging
- Crystal monochromators
- Electronic band structures

Further reading

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Single-crystal X-ray diffraction

1. REDIRECT X-ray crystallography

This is a redirect from a title with another method of capitalisation. It leads to the title in accordance with the Wikipedia naming conventions for capitalisation, and can help writing, searching, and international language issues.

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For more information, see [Category:Redirects from other capitalisations](#).

Macromolecules

1. REDIRECT Macromolecule

Proteins

1. REDIRECT Protein

This is a redirect from a plural word to the singular equivalent.

You may use the aliased template `{{R to singular}}` to accomplish the same end.

This redirect link is used for convenience, usually for plurals that do not follow simple conventions. In many cases, it is preferable to add the plural directly after the link (that is, `[[link]]s`). However, do not replace these redirected links with a simpler link unless the page is updated for another reason (see [Wikipedia:Redirect#Do not "fix" links to redirects that are not broken](#)).

For more information, follow the category link.

Powder diffraction

Powder diffraction is a scientific technique using X-ray, neutron, or electron diffraction on powder or microcrystalline samples for structural characterization of materials.^[2]

Explanation

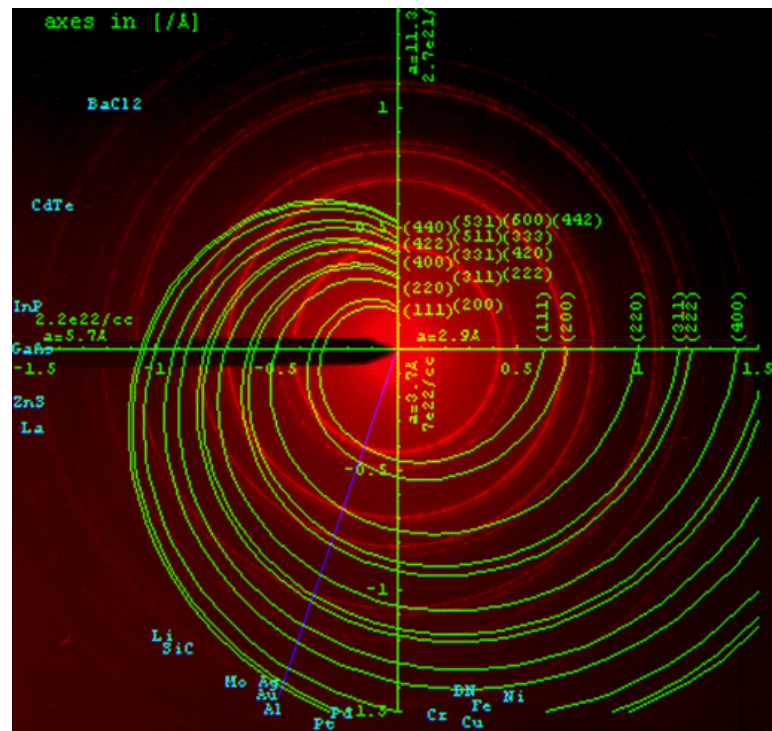
Ideally, every possible crystalline orientation is represented equally in a powdered sample. The resulting orientational averaging causes the three dimensional reciprocal space that is studied in single crystal diffraction to be projected onto a single dimension. The three dimensional space can be described with (reciprocal) axes x^* , y^* and z^* or

alternatively in spherical coordinates q , φ^* , χ^* . In powder diffraction intensity is homogeneous over φ^* and χ^* and only q remains as an important measurable quantity. In practice, it is sometimes necessary to rotate the sample orientation to eliminate the effects of texturing and achieve true randomness.

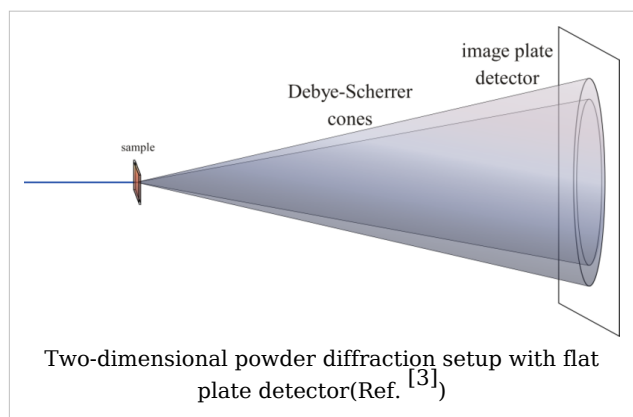
When the scattered radiation is collected on a flat plate detector the rotational averaging leads to smooth diffraction rings around the beam axis rather than the discrete Laue spots as observed for single crystal diffraction. The angle between the beam axis and the ring is called the *scattering angle* and in X-ray crystallography always denoted as 2θ . (In scattering of *visible* light the convention is usually to call it θ). In accordance with Bragg's law, each ring corresponds to a particular reciprocal lattice vector G in the sample crystal. This leads to the definition of the scattering vector as:

$$G = q = 2k \sin(\theta) = 4\pi \sin(\theta) / \lambda.$$

Powder diffraction data are usually presented as a *diffractogram* in which the diffracted intensity I is shown as function either of the scattering angle 2θ or as a function of the



Electron powder pattern (red) of an Al film with an fcc spiral overlay (green) and a line of intersections (blue) that determines lattice parameter.^[1]



scattering vector q . The latter variable has the advantage that the diffractogram no longer depends on the value of the wavelength λ . The advent of synchrotron sources has widened the choice of wavelength considerably. To facilitate comparability of data obtained with different wavelengths the use of q is therefore recommended and gaining acceptability.

An instrument dedicated to perform powder measurements is called a **powder diffractometer**.

Uses

Relative to other methods of analysis, powder diffraction allows for rapid, non-destructive analysis of multi-component mixtures without the need for extensive sample preparation.^[4] This gives laboratories around the world the ability to quickly analyse unknown materials and perform materials characterization in such fields as metallurgy, mineralogy, forensic science, archeology, condensed matter physics, and the biological and pharmaceutical sciences. Identification is performed by comparison of the diffraction pattern to a known standard or to a database such as the International Centre for Diffraction Data's Powder Diffraction File (PDF) or the Cambridge Structural Database (CSD). Advances in hardware and software, particularly improved optics and fast detectors, have dramatically improved the analytical capability of the technique, especially relative to the speed of the analysis. The fundamental physics upon which the technique is based provides high precision and accuracy in the measurement of interplanar spacings, sometimes to fractions of an Ångström, resulting in authoritative identification frequently used in patents, criminal cases and other areas of law enforcement. The ability to analyze multiphase materials also allows analysis of how materials interact in a particular matrix such as a pharmaceutical tablet, a circuit board, a mechanical weld, a geologic core sampling, cement and concrete, or a pigment found in an historic painting. The method has been historically used for the identification and classification of minerals, but it can be used for any materials, even amorphous ones, so long as a suitable reference pattern is known or can be constructed.

Phase identification

The most widespread use of powder diffraction is in the identification and characterization of crystalline solids, each of which produces a distinctive diffraction pattern. Both the positions (corresponding to lattice spacings) and the relative intensity of the lines are indicative of a particular phase and material, providing a "fingerprint" for comparison. A multi-phase mixture, *e.g.* a soil sample, will show more than one pattern superposed, allowing for determination of relative concentration.

J.D. Hanawalt, an analytical chemist who worked for Dow Chemical in the 1930s, was the first to realize the analytical potential of creating a database. Today it is represented by the Powder Diffraction File (PDF) of the International Centre for Diffraction Data (formerly Joint Committee for Powder Diffraction Studies). This has been made searchable by computer through the work of global software developers and equipment manufacturers. There are now over 550,000 reference materials in the 2006 Powder Diffraction File Databases, and these databases are interfaced to a wide variety of diffraction analysis software and distributed globally. The Powder Diffraction File contains many subfiles, such as minerals, metals and alloys, pharmaceuticals, forensics, excipients, superconductors, semiconductors *etc.*, with large collections of organic, organometallic and inorganic reference materials.

Crystallinity

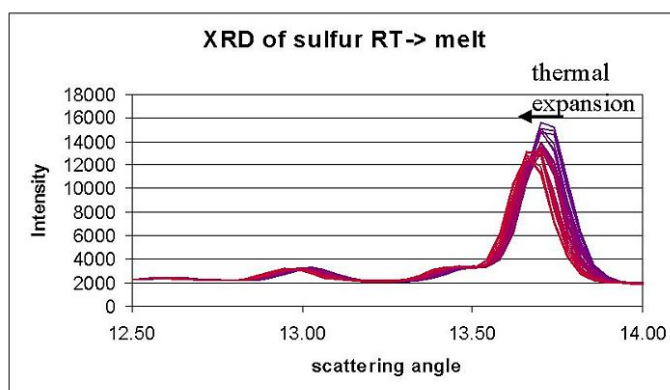
In contrast to a crystalline pattern consisting of a series of sharp peaks, amorphous materials (liquids, glasses etc.) produce a broad background signal. Many polymers show semicrystalline behavior, *i.e.* part of the material forms an ordered crystallite by folding of the molecule. One and the same molecule may well be folded into two different crystallites and thus form a tie between the two. The tie part is prevented from crystallizing. The result is that the crystallinity will never reach 100%. Powder XRD can be used to determine the crystallinity by comparing the integrated intensity of the background pattern to that of the sharp peaks. Values obtained from powder XRD are typically comparable but not quite identical to those obtained from other methods such as DSC.

Lattice parameters

The position of a diffraction peak is *independent* of the atomic positions within the cell and entirely determined by the size and shape of the unit cell of the crystalline phase. Each peak represents a certain lattice plane and can therefore be characterized by a Miller index. If the symmetry is high, e.g. cubic or hexagonal it is usually not too hard to identify the index of each peak, even for an unknown phase. This is particularly important in solid-state chemistry, where one is interested in finding and identifying new materials. Once a pattern has been indexed, this characterizes the reaction product and identifies it as a new solid phase. Indexing programs exist to deal with the harder cases, but if the unit cell is very large and the symmetry low (triclinic) success is not always guaranteed.

Expansion tensors, bulk modulus

Cell parameters are somewhat temperature and pressure dependent. Powder diffraction can be combined with *in situ* temperature and pressure control. As these thermodynamic variables are changed, the observed diffraction peaks will migrate continuously to indicate higher or lower lattice spacings as the unit cell distorts. This allows for measurement of such quantities as the thermal expansion tensor and the isothermal bulk modulus, as well determination of the full equation of state of the material.



Thermal expansion of a sulfur powder

Phase transitions

At some critical set of conditions, for example 0 °C for water at 1 atm, a new arrangement of atoms or molecules may become stable, leading to a phase transition. At this point new diffraction peaks will appear or old ones disappear according to the symmetry of the new phase. If the material melts to an isotropic liquid, all sharp lines will disappear and be replaced by a broad amorphous pattern. If the transition produces another crystalline phase, one set of lines will suddenly be replaced by another set. In some cases however lines will split or coalesce, e.g. if the material undergoes a continuous, second order phase transition. In such cases the symmetry may change because the existing structure is *distorted* rather than replaced by a completely different one. E.g. the diffraction peaks for the lattice planes (100) and (001) can be found at two different values of q for a tetragonal phase, but if the symmetry becomes cubic the two peaks will come to coincide.

Crystal structure refinement and determination

Crystal structure determination from powder diffraction data is extremely challenging due to the overlap of reflections in a powder experiment. The crystal structures of known materials can be refined, i.e. as a function of temperature or pressure, using the Rietveld method. The Rietveld method is a so-called full pattern analysis technique. A crystal structure, together with instrumental and microstructural information is used to generate a theoretical diffraction pattern that can be compared to the observed data. A least squares procedure is then used to minimise the difference between the calculated pattern and each point of the observed pattern by adjusting model parameters. Techniques to determine unknown structures from powder data do exist, but are somewhat specialised.^[5]

Size and strain broadening

There are many factors that determine the width B of a diffraction peak. These include:

1. instrumental factors
2. the presence of defects to the perfect lattice
3. differences in strain in different grains
4. the size of the crystallites

It is often possible to separate the effects of size and strain. Where size broadening is independent of q ($K=1/d$), strain broadening increases with increasing q -values. In most cases there will be both size and strain broadening. It is possible to separate these by combining the two equations in what is known as the Hall-Williamson method:

$$B \cdot \cos\theta = \frac{k\lambda}{D} + \eta \sin\theta$$

Thus, when we plot $B \cdot \cos\theta$ vs. $\sin\theta$ we get a straight line with slope η and intercept $\frac{k\lambda}{D}$.

The expression is a combination of the Debye-Scherrer formula for size broadening and the Stokes and Wilson expression for strain broadening. The value of η is the strain in the crystallites, the value of D represents the size of the crystallites. The constant k is typically close to unity and ranges from 0.8-1.39.

Comparison of X-ray and Neutron Scattering

X-ray photons scatter by interaction with the electron cloud of the material, neutrons are scattered by the nuclei. This means that, in the presence of heavy atoms with many electrons, it may be difficult to detect light atoms by X-ray diffraction. In contrast, the neutron scattering length of most atoms are approximately equal in magnitude. Neutron diffraction techniques may therefore be used to detect light elements such as oxygen or hydrogen in combination with heavy atoms. The neutron diffraction technique therefore has obvious applications to problems such as determining oxygen displacements in materials like high temperature superconductors and ferroelectrics, or to hydrogen bonding in biological systems.

A further complication in the case of neutron scattering from hydrogenous materials is the strong incoherent scattering of hydrogen (80.27(6) barn). This leads to a very high background in neutron diffraction experiments, and may make structural investigations impossible. A common solution is deuteration, i.e. replacing the 1-H atoms in the sample with deuterium (2-H). The incoherent scattering length of deuterium is much smaller (2.05(3) barn) making structural investigations significantly easier. However, in some systems, replacing hydrogen with deuterium may alter the structural and dynamic properties of interest.

As neutrons also have a magnetic moment, they are additionally scattered by any magnetic moments in a sample. In the case of long range magnetic order, this leads to the appearance of new Bragg reflections. In most simple cases, powder diffraction may be used to determine the size of the moments and their spatial orientation.

Aperiodically-arranged clusters

Predicting the scattered intensity in powder diffraction patterns from gases, liquids, and randomly-distributed nano-clusters in the solid state^[6] is (to first order) done rather elegantly with the Debye scattering equation^[7]:

$$I_{\text{powder}}(q) = \sum_{i=1}^N \sum_{j=1}^N f_i(q) f_j(q) \frac{\sin(qr_{ij})}{qr_{ij}},$$

where the magnitude of the scattering vector q is in reciprocal lattice distance units, N is the number of atoms, $f_i(q)$ is the atomic scattering factor for atom i and scattering vector q , while r_{ij} is the distance between atom i and atom j . One can also use this to predict the effect of nano-crystallite shape on detected diffraction peaks, even if in some directions the cluster is only one atom thick.

Devices

Cameras

The simplest cameras for X-ray powder diffraction consist of a small capillary and either a flat plate detector (originally a piece of X-ray film, now more and more a flat-plate detector or a CCD-camera) or a cylindrical one (originally a piece of film in a cookie-jar, now more and more a bent position sensitive detector). The two types of cameras are known as the Laue and the Debye-Scherrer camera.

In order to ensure complete powder averaging, the capillary is usually spun around its axis.

For neutron diffraction vanadium cylinders are used as sample holders. Vanadium has a negligible absorption and coherent scattering cross section for neutrons and is hence nearly invisible in a powder diffraction experiment. Vanadium does however have a considerable incoherent scattering cross section which may cause problems for more sensitive techniques such as neutron inelastic scattering.

A later development in X-ray cameras is the Guinier camera. It is built around a *focusing* bent crystal monochromator. The sample is usually placed in the focusing beam., e.g. as a dusting on a piece of sticky tape. A cylindrical piece of film (or electronic multichannel detector) is put on the focusing circle, but the incident beam prevented from reaching the detector to prevent damage from its high intensity.

Diffractometers

Diffractionometers can be operated both in transmission and in reflection configurations. The reflection one is more common. The powder sample is filled in a small disc like container and its surface carefully flattened. The disc is put on one axis of the diffractometer and tilted by an angle θ while a detector (scintillation counter) rotates around it on an arm at twice this angle. This configuration is known under the name Bragg-Brentano.

Another configuration is the theta-theta configuration in which the sample is stationary while the X-ray tube and the detector are rotated around it. The angle formed between the tube and the detector is 2θ . This configuration is most convenient for loose powders.

The availability of position sensitive detectors and CCD-cameras is making this type of equipment more and more obsolete.

Neutron diffraction

Sources that produce a neutron beam of suitable intensity and speed for diffraction are only available at a small number of research reactors and spallation sources in the world. Angle dispersive (fixed wavelength) instruments typically have a battery of individual detectors arranged in a cylindrical fashion around the sample holder, and can therefore collect scattered intensity simultaneously on a large 2θ range. Time of flight instruments normally have a small range of banks at different scattering angles which collect data at varying resolutions.

X-ray tubes

Laboratory X-ray diffraction equipment relies on the use of an X-ray tube, which is used to produce the X-rays.

For more on how X-ray tubes work, see for example here ^[8] or X-ray.

The most commonly used laboratory X-ray tube uses a Copper anode, but Cobalt, Molybdenum are also popular. The wavelength in nm varies for each source:

Element	K α (weight average)	K α 2 (strong)	K α 1 (very strong)	K β (weak)
Cr	0.229100	0.229361	0.228970	0.208487
Fe	0.193736	0.193998	0.193604	0.175661
Co	0.179026	0.179285	0.178897	0.162079
Cu	0.154184	0.154439	0.154056	0.139222

Mo	0.071073	0.071359	0.070930	0.063229
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Other sources

In house applications of X-ray diffraction has always been limited to the relatively few wavelengths shown in the table above. The available choice was much needed because the combination of certain wavelengths and certain elements present in a sample can lead to strong fluorescence which increases the background in the diffraction pattern. A notorious example is the presence of iron in a sample when using copper radiation. In general elements just below the anode element in the period system need to be avoided.

Another limitation is that the intensity of traditional generators is relatively low, requiring lengthy exposure times and precluding any time dependent measurement. The advent of synchrotron sources has drastically changed this picture and caused powder diffraction methods to enter a whole new phase of development. Not only is there a much wider choice of wavelengths available, the high brilliance of the synchrotron radiation makes it possible to observe changes in the pattern during chemical reactions, temperature ramps, changes in pressure and the like.

The tunability of the wavelength also makes it possible to observe anomalous scattering effects when the wavelength is chosen close to the absorption edge of one of the elements of the sample.

Neutron diffraction has never been an in house technique because it requires the availability of an intense neutron beam only available at a nuclear reactor. Typically the available neutron flux, and the weak interaction between neutrons and matter, require relative large samples.

Advantages and disadvantages

Although it possible to solve crystal structures from powder X-ray data alone, its single crystal analogue is a far more powerful technique for structure determination. This is directly related to the fact that much information is lost by the collapse of the 3D space onto a 1D axis. Nevertheless powder X-ray diffraction is a powerful and useful technique in its own right. It is mostly used to characterize and identify *phases* rather than solving structures.

The great advantages of the technique are:

- simplicity of sample preparation
- rapidity of measurement
- the ability to analyse mixed phases, e.g. soil samples

By contrast growth and mounting of large single crystals is notoriously difficult. In fact there are many materials for which despite many attempts it has not proven possible to obtain single crystals. Many materials are readily available with sufficient microcrystallinity for powder diffraction, or samples may be easily ground from larger crystals. In the field of solid-state chemistry that often aims at synthesizing *new* materials, single crystals thereof are typically not immediately available. Powder diffraction is therefore one of the most powerful methods to identify and characterize new materials in this field.

Particularly for neutron diffraction, which requires larger samples than X-Ray Diffraction due to a relatively weak scattering cross section, the ability to use large samples can be

critical, although new more brilliant neutron sources are being built that may change this picture.

Since all possible crystal orientations are measured simultaneously, collection times can be quite short even for small and weakly scattering samples. This is not merely convenient, but can be essential for samples which are unstable either inherently or under X-ray or neutron bombardment, or for time-resolved studies. For the latter it is desirable to have a strong radiation source. The advent of synchrotron radiation and modern neutron sources has therefore done much to revitalize the powder diffraction field because it is now possible to study temperature dependent changes, reaction kinetics and so forth by means of time dependent powder diffraction.

See also

- Bragg diffraction
- Condensed Matter Physics
- Crystallography
- Crystallographic database
- Electron crystallography
- Electron diffraction
- Materials science
- Metallurgy
- Neutron diffraction
- Neutron crystallography
- Solid state chemistry
- Texture (crystalline)
- X-ray crystallography
- X-ray diffraction
- X-ray scattering techniques

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 - [2] B.D. Cullity Elements of X-ray Diffraction Addison Wesley Mass. 1978
 - [3] <http://dx.doi.org/10.1080/07303300310001634952>
 - [4] cf. Chapter 14 of Elements of X-ray diffraction, B.D. Cullity, Addison-Wesley, 2nd ed. 1977 ISBN 0-201-01174-3
 - [5] Structure determination from powder diffraction data IUCr Monographs on crystallography, Edt. W.I.F. David, K. Shankland, L.B. McCusker and Ch. Baerlocher. 2002. Oxford Science publications ISBN 0-19-850091-2
 - [6] B. E. Warren (1969/1990) *X-ray diffraction* (Addison-Wesley, Reading MA/Dover, Mineola NY).
 - [7] P. Debye (1915) *Ann. Physik* **46**:809.
 - [8] <http://hyperphysics.phy-astr.gsu.edu/Hbase/quantum/xtube.html>
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Further Reading

- Pecharsky, Vitalij; Zavalij, Peter (2008). *Fundamentals of Powder Diffraction and Structural Characterization of Materials* (2nd ed.). Springer. ISBN 0-387-09578-0.
- Cullity, B.D. (1978). *Elements of X-Ray Diffraction* (2nd ed.). Reading, Massachusetts: Addison-Wesley Publishing Company. ISBN 0-534-55396-6.
- Young, R.A., ed (1993). *The Rietveld Method*. Oxford: Oxford University Press & International Union of Crystallography. ISBN 0-19-855577-6.

External links

- International Centre for Diffraction Data (<http://www.icdd.com>)
- The Area Diffraction Machine - software to analyze powder diffraction data (<http://areadiffractionmachine.googlecode.com>)
- Nanoscale Interdisciplinary Research Team (<http://nirt.pa.msu.edu/>)

International Centre for Diffraction Data

The **International Centre for Diffraction Data (ICDD)** maintains a database of powder diffraction patterns, the **Powder Diffraction File (PDF)**, including the d-spacings (related to angle of diffraction) and relative intensities of observable diffraction peaks. Patterns may be experimentally determined, or computed based on crystal structure and Bragg's law. It is most often used to identify substances based on x-ray diffraction data, and is designed for use with a diffractometer. The database is exhaustive, over 500,000 entries as of 2006; computer algorithms allow rapid peak matching. The organization was founded in 1941 as the **Joint Committee on Powder Diffraction Standards (JCPDS)**. In 1978, the name of the organization was changed to the current name to highlight the global commitment of this scientific endeavor.

The ICDD is a nonprofit scientific organization, founded by, and dedicated to scientists working in the field of X-ray analysis and materials characterization. The ICDD produces materials databases, characterization tools, and educational materials, as well as organizing and supporting global workshops, clinics and conferences. It provides the scientific community with the tools required for X-ray analysis while continuing to educate and inform current and future scientists in the field.

Products and services of the ICDD include the Powder Diffraction File databases, educational workshops, clinics and symposia as well as sponsorship of the Denver X-ray Conference. The ICDD also publishes the journals *Advances in X-ray Analysis* and *Powder Diffraction*.

As mentioned above, the Powder Diffraction File contains powder diffraction data and is designed to work with a diffractometer to identify unknown materials. With every entry, the database also contains bibliographic references, chemistry descriptions, structural classifications, crystallographic and physical properties.

See also

- powder diffraction
- crystallography

External links

- International Centre for Diffraction Data website ^[1] -- Information about the ICDD and its products and services
- History, contents & use of the PDF ^[2]
- Advances in X-ray Analysis ^[3] -- Technical articles on x-ray methods and analyses

References

[1] <http://www.icdd.com>

[2] <http://www.icdd.com/products/ICDD.PDF>

[3] http://www.icdd.com/resources/axasearch/AXA_login.php

Scherrer Equation

1. REDIRECT Shape factor (X-ray diffraction)

Scattering techniques

Small angle X-ray scattering (SAXS)

1. REDIRECT Small-angle X-ray scattering

X-ray reflectivity

X-ray reflectivity sometimes known as **X-ray specular reflectivity**, **X-ray reflectometry**, or **XRR**, is a surface-sensitive analytical technique used in chemistry, physics, and materials science to characterize surfaces, thin films and multilayers.^{[1] [2] [3] [4]} It is related to the complementary techniques of neutron reflectometry and ellipsometry.

The basic idea behind the technique is to reflect a beam of x-rays from a flat surface and to then measure the intensity of x-rays reflected in the specular direction (reflected angle equal to incident angle). If the interface is not perfectly sharp and smooth then the reflected intensity will deviate from that predicted by the law of Fresnel reflectivity. The deviations can then be analyzed to obtain the density profile of the interface normal to the surface.

The technique appears to have first been applied to x-rays by Professor Lyman G. Parratt of Cornell University in an article published in the physical review in 1954.^[5] Parratt's initial work explored the surface of copper-coated glass, but since that time the technique has been extended to a wide range of both solid and liquid interfaces.

The basic mathematical relationship which describes specular reflectivity is fairly straightforward. When an interface is not perfectly sharp, but has an average electron density profile given by $\rho_e(z)$, then the x-ray reflectivity can be approximated by:

$$R(Q)/R_F(Q) = \left| \frac{1}{\rho_\infty} \int_{-\infty}^{\infty} e^{iQz} \rho_e(z) dz \right|^2$$

Here $R(Q)$ is the reflectivity, $Q = 4\pi \sin(\theta)/\lambda$, λ is the x-ray wavelength, ρ_∞ is the density deep within the material and θ is the angle of incidence. Typically one can then use this formula to compare parametrized models of the average density profile in the z-direction with the measured x-ray reflectivity and then vary the parameters until the theoretical profile matches the measurement.

For films with multiple layers, X-ray reflectivity may show oscillations with wavelength, analogous to the Fabry-Pérot effect. These oscillations can be used to infer layer thicknesses and other properties, for example using the Abeles matrix formalism.

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- [1] Holy, V. et al. *Phys. Rev. B.* **47**, 15896 (1993).
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- [3] J.Daillant, A.Gibaud, *X-Ray and Neutron Reflectivity: Principles and Applications*. Springer, (1999).
- [4] M.Tolan, *X-Ray Scattering from Soft-Matter Thin Films*, Springer, (1999)
- [5] L. G. Parratt, *Phys. Rev.* **95**, 359 (1954).

Wide angle X-ray scattering

Wide angle X-ray scattering (WAXS) or Wide angle X-ray diffraction (WAXD) is an X-ray diffraction technique that is often used to determine the crystalline structure of polymers. This technique specifically refers to the analysis of Bragg Peaks scattered to wide angles, which (by Bragg's law) implies that they are caused by sub-nanometer sized structures.

Wide angle x-ray scattering is the same technique as Small-Angle X-ray Scattering (SAXS) only the distance from sample to the detector is shorter and thus diffraction maxima at larger angles are observed.

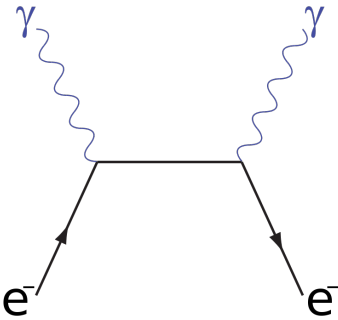
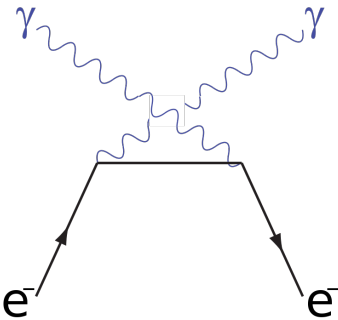
The technique is a time-honored but a somewhat out-of-favor technique for the determination of degree of crystallinity of polymer samples. A diffraction technique for polycrystalline films where only crystallites diffract which are parallel to the substrate surface. The diffraction pattern generated allows to determine the chemical composition or phase composition of the film, the texture of the film (preferred alignment of crystallites), the crystallite size and presence of film stress. According to this method the sample is scanned in a wide angle X-ray goniometer, and the scattering intensity is plotted as a function of the 2θ angle. X ray diffraction is a non destructive method of characterization of solid materials. When X-rays are directed in solids they will scatter in predictable patterns based upon the internal structure of the solid. A crystalline solid consists of regularly spaced atoms (electrons) that can be described by imaginary planes. The distance between these planes is called the d-spacing. The intensity of the d-space pattern is directly proportional to the number of electrons (atoms) that are found in the imaginary planes. Every crystalline solid will have a unique pattern of d-spacings (known as the powder pattern), which is a “finger print” for that solid. In fact solids with the same chemical composition but different phases can be identified by their pattern of d-spacings.

See also

- X-ray crystallography
-

Inelastic scattering

Compton scattering

Compton scattering	
Feynman diagrams	
<p>s-channel</p> 	
<p>u-channel</p> 	
Light-matter interaction	
Low energy phenomena	Photoelectric effect
Mid-energy phenomena	Compton scattering
High energy phenomena	Pair production

In physics, **Compton scattering** or the **Compton effect** is the decrease in energy (increase in wavelength) of an X-ray or gamma ray photon, when it interacts with matter. Because of the change in photon energy, it is an inelastic scattering process. **Inverse Compton scattering** also exists, where the photon gains energy (decreasing in wavelength) upon interaction with matter. The amount the wavelength changes by is called the **Compton shift**. Although nuclear compton scattering exists^[1], Compton scattering usually refers to the interaction involving only the electrons of an atom. The Compton effect was observed by Arthur Holly Compton in 1923 and further verified by his graduate student Y. H. Woo in the years following. Arthur Compton earned the 1927 Nobel Prize in Physics for the discovery.

The effect is important because it demonstrates that light cannot be explained purely as a wave phenomenon. Thomson scattering, the classical theory of an electromagnetic wave scattered by charged particles, cannot explain low intensity shift in wavelength (Classically,

light of sufficient intensity for the electric field to accelerate a charged particle to a relativistic speed will cause radiation-pressure recoil and an associated Doppler shift of the scattered light, but the effect would become arbitrarily small at sufficiently low light intensities regardless of wavelength.). Light must behave as if it consists of particles in order to explain the low-intensity Compton scattering. Compton's experiment convinced physicists that light can behave as a stream of particle-like objects (quanta) whose energy is proportional to the frequency.

The interaction between electrons and high energy photons ($\sim\text{keV}$) results in the electron being given part of the energy (making it recoil), and a photon containing the remaining energy being emitted in a different direction from the original, so that the overall momentum of the system is conserved. If the photon still has enough energy left, the process may be repeated. In this scenario, the electron is treated as free or loosely bound. Experimental verification of momentum conservation in individual Compton scattering processes by Bothe and Geiger as well as by Compton and Simon has been important in disproving the BKS theory.

If the photon is of lower energy, but still has sufficient energy (in general a few eV, right around the energy of visible light), it can eject an electron from its host atom entirely (a process known as the photoelectric effect), instead of undergoing Compton scattering. Higher energy photons ($\sim\text{MeV}$) may be able to bombard the nucleus and cause an electron and a positron to be formed, a process called pair production.

The Compton shift formula

Compton used a combination of three fundamental formulas representing the various aspects of classical and modern physics, combining them to describe the quantum behavior of light.

- Light as a particle, as noted previously in the photoelectric effect.
- Relativistic dynamics: special theory of relativity
- Trigonometry: law of cosines

The final result gives us the **Compton scattering equation**:

$$\lambda' - \lambda = \frac{h}{m_e c} (1 - \cos \theta)$$

where

λ is the wavelength of the photon **before** scattering,

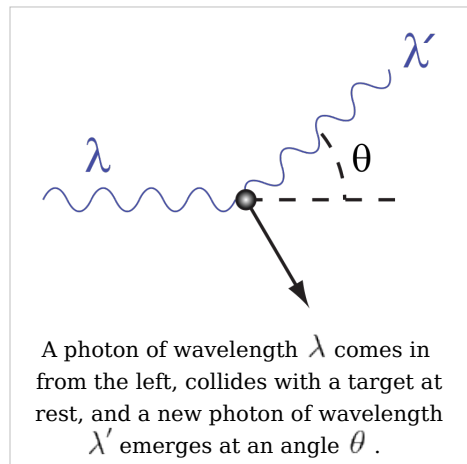
λ' is the wavelength of the photon **after** scattering,

m_e is the mass of the electron,

θ is the angle by which the photon's heading changes (between 0° and 180°),

h is Planck's constant, and

c is the speed of light.



$\frac{h}{m_e c} = 2.43 \times 10^{-12}$ m is known as the Compton wavelength. $\lambda' - \lambda$ can be between 0 (for $\theta = 0^\circ$) and two times the Compton wavelength (for $\theta = 180^\circ$).

Derivation

Begin with conservation of energy and conservation of momentum:

$$E_\gamma + E_e = E_{\gamma'} + E_{e'} \quad (1)$$

$$\vec{p}_\gamma = \vec{p}_{\gamma'} + \vec{p}_{e'} \quad (2)$$

where

E_γ and p_γ are the energy and momentum of the photon and
 E_e and p_e are the energy and momentum of the electron.

Solving (Part 1)

Now we fill in for the energy part:

$$E_\gamma + E_e = E_{\gamma'} + E_{e'}$$

$$hf + mc^2 = hf' + \sqrt{(p_{e'}c)^2 + (mc^2)^2}$$

The square of the second equation gives an equation for $p_{e'}$:

$$p_{e'}^2 c^2 = (hf + mc^2 - hf')^2 - m^2 c^4 \quad (3)$$

Solving (Part 2)

Rearrange equation (2)

$$\vec{p}_{e'} = \vec{p}_\gamma - \vec{p}_{\gamma'}$$

and square it to see

$$p_{e'}^2 = (\vec{p}_\gamma - \vec{p}_{\gamma'}) \cdot (\vec{p}_\gamma - \vec{p}_{\gamma'})$$

$$p_{e'}^2 = p_\gamma^2 + p_{\gamma'}^2 - 2\vec{p}_\gamma \cdot \vec{p}_{\gamma'}$$

$$p_{e'}^2 = p_\gamma^2 + p_{\gamma'}^2 - 2|p_\gamma||p_{\gamma'}|\cos(\theta)$$

Energy and momentum of photons are connected by the relativistic equation $p_\gamma = \frac{E_\gamma}{c}$, so
 $E_\gamma^2 = p_\gamma^2 c^2 = (hf)^2$.

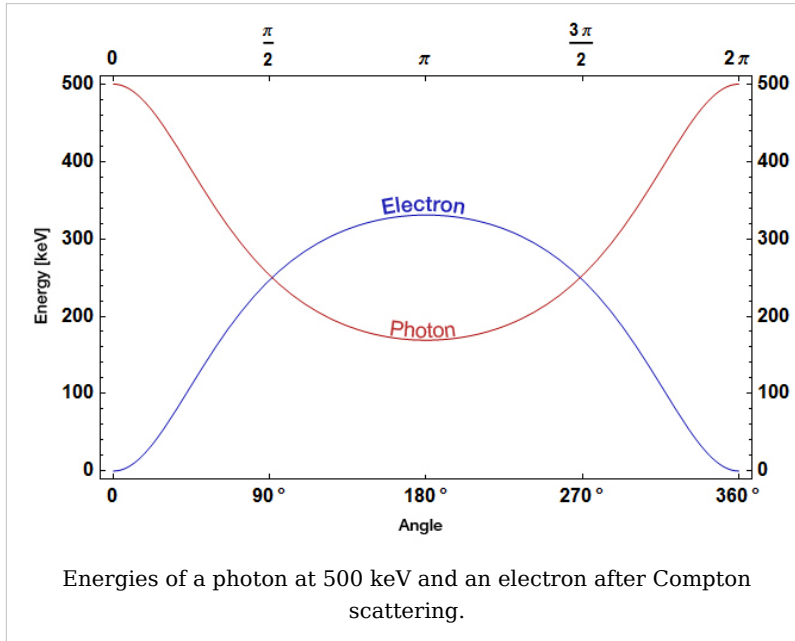
Therefore, multiplying by c^2 , we have also

$$p_{e'}^2 c^2 = (hf)^2 + (hf')^2 - 2(hf)(hf')\cos\theta \quad (4)$$

Putting it together

Now we have the two equations (3 & 4) for $p_e^2 c^2$, which we equate:

$$(hf)^2 + (hf')^2 - 2h^2 f f' \cos \theta = (hf + mc^2 - hf')^2 - m^2 c^4$$



$$-2h^2 f f' \cos \theta = -2h^2 f f' + 2hfmc^2 - 2hf'mc^2.$$

Combining two terms, this becomes:

$$2h^2 f f' (1 - \cos(\theta)) = 2hfmc^2 - 2hf'mc^2.$$

After dividing both sides by $(2hf f' mc^2)$, we get:

$$\frac{h}{mc^2} (1 - \cos \theta) = \frac{1}{f'} - \frac{1}{f}$$

This is equivalent to the **Compton scattering equation**, but it is usually written in terms of wavelength rather than frequency. To make that switch use

$$f = \frac{c}{\lambda}$$

so that finally,

$$\lambda' - \lambda = \frac{h}{mc} (1 - \cos \theta)$$

Applications

Compton scattering

Compton scattering is of prime importance to radiobiology, as it happens to be the most probable interaction of high energy X rays with atomic nuclei in living beings and is applied in radiation therapy.^[2]

In material physics, Compton scattering can be used to probe the wave function of the electrons in matter in the momentum representation.

Compton scattering is an important effect in gamma spectroscopy which gives rise to the Compton edge, as it is possible for the gamma rays to scatter out of the detectors used. Compton suppression is used to detect stray scatter gamma rays to counteract this effect.

Inverse Compton scattering

Inverse Compton scattering is important in astrophysics. In X-ray astronomy, the accretion disk surrounding a black hole is believed to produce a thermal spectrum. The lower energy photons produced from this spectrum are scattered to higher energies by relativistic electrons in the surrounding corona. This is believed to cause the power law component in the X-ray spectra (0.2-10 keV) of accreting black holes.

The effect is also observed when photons from the cosmic microwave background move through the hot gas surrounding a galaxy cluster. The CMB photons are scattered to higher energies by the electrons in this gas, resulting in the Sunyaev-Zel'dovich effect. Observations of the Sunyaev-Zel'dovich effect provide a nearly redshift-independent means of detecting galaxy clusters.

See also

- Thomson scattering
- Klein-Nishina formula
- Photoelectric effect
- Pair production
- Timeline of cosmic microwave background astronomy
- Peter Debye
- Walther Bothe
- List of astronomical topics
- List of physics topics
- Washington University in St. Louis (Site of discovery)

Notes

- [1] P Christillin (1986). "<http://www.iop.org/EJ/abstract/0305-4616/12/9/008>[Nuclear Compton scattering". *J. Phys. G: Nucl. Phys.* **12**: 837–851. doi: 10.1088/0305-4616/12/9/008 (<http://dx.doi.org/10.1088/0305-4616/12/9/008>). <http://www.iop.org/EJ/abstract/0305-4616/12/9/008>.
- [2] Camphausen KA, Lawrence RC. "Principles of Radiation Therapy" (<http://www.cancernetwork.com/cancer-management-11/chapter02/article/10165/1399960>) in Pazdur R, Wagman LD, Camphausen KA, Hoskins WJ (Eds) *Cancer Management: A Multidisciplinary Approach* (<http://www.cancernetwork.com/cancer-management-11/>). 11 ed. 2008.

Further reading

- S. Chen; H. Avakian, V. Burkert, P. Eugenio, the CLAS collaboration (2006). "<http://arxiv.org/abs/hep-ex/0605012>[Measurement of Deeply Virtual Compton Scattering with a Polarized Proton Target". *Physical Review Letters* **97**: 072002. doi: 10.1103/PhysRevLett.97.072002 (<http://dx.doi.org/10.1103/PhysRevLett.97.072002>). <http://arxiv.org/abs/hep-ex/0605012>.
- Compton, Arthur H. (May 1923). "http://prola.aps.org/abstract/PR/v21/i5/p483_1|A Quantum Theory of the Scattering of X-Rays by Light Elements". *The Physical Review* **21** (5): 483–502. doi: 10.1103/PhysRev.21.483 (<http://dx.doi.org/10.1103/PhysRev.21.483>). http://prola.aps.org/abstract/PR/v21/i5/p483_1. (the original 1923 paper on

the AIP website)

External links

- BIGS animation (http://www.bigs.de/BLH/en/index.php?option=com_content&view=category&layout=blog&id=85&Itemid=253) compton effect
- Compton Scattering (<http://hyperphysics.phy-astr.gsu.edu/Hbase/quantum/comptint.html>) - Georgia State University
- Compton Scattering Data (<http://hyperphysics.phy-astr.gsu.edu/Hbase/quantum/compdat.html#c1>) - Georgia State University

X-ray Raman scattering

X-ray Raman scattering (XRS) is non-resonant inelastic scattering of x-rays from core electrons. It is analogous to Raman scattering, which is a largely-used tool in optical spectroscopy, with the difference being that the wavelengths of the exciting photons fall in the x-ray regime and the corresponding excitations are from deep core electrons.

XRS is an element-specific spectroscopic tool for studying the electronic structure of matter. In particular, it probes the excited-state density of states (DOS) of an atomic species in a sample. ^[1]

Description

XRS is an inelastic x-ray scattering process, in which a high-energy x-ray photon gives energy to a core electron, exciting it to an unoccupied state. The process is in principle analogous to x-ray absorption (XAS), but the *energy transfer* plays the role of the x-ray photon *energy absorbed* in x-ray absorption, exactly as in Raman scattering in optics vibrational low-energy excitations can be observed by studying the spectrum of light scattered from a molecule.

Because the energy (i.e. wavelength) of the probing x-ray can be chosen freely and is usually in the hard x-ray regime, certain constraints of soft x-rays in the studies of electronic structure of the material are overcome. For example, soft x-ray studies may be surface sensitive and they require a vacuum environment. This makes studies of e.g. many liquids impossible using soft x-ray absorption. One of the most notable applications in which x-ray Raman scattering is superior to soft x-ray absorption is the study of soft x-ray absorption edges in high pressure. Whereas high-energy x rays may pass through a high-pressure apparatus like a diamond anvil cell and reach the sample inside the cell, soft x-rays would be absorbed by the cell itself.

History

In his report of finding of a new type of scattering, Chandrasekhara Venkata Raman proposed that a similar effect should be found also in the x-ray regime. Around the same time, B. Davis and D. Mitchell reported in 1928 on the fine-structure of the scattered radiation from graphite and noted that they had lines that seemed to be in agreement with carbon K shell energy. Several researchers attempted similar experiments in the late 1920s and early 1930s but the results could not always be confirmed. Often the first unambiguous

observations of the XRS effect is credited to K. Das Gupta (reported findings 1959) and Tadasu Suzuki (reported 1964). It was soon realized that the XRS peak in solids was broadened by the solid-state effects and it appeared as a band, with a shape similar to that of a XAS spectrum. The potential of the technique was limited until modern synchrotron light sources became available. This is due to the very small XRS probability of the incident photons, requiring radiation with a very high intensity. Today, the XRS technique is rapidly growing in importance. It can be used to study near-edge x-ray absorption fine structure (NEXAFS or XANES) as well as extended x-ray absorption fine structure (EXAFS).

Brief theory of XRS

XRS belongs to the class of non-resonant inelastic x-ray scattering, which has a cross section of

$$\frac{d^2\sigma}{d\Omega dE} = \left(\frac{d\sigma}{d\Omega}\right)_{\text{Th}} \times S(q, E).$$

Here, $(d\sigma/d\Omega)_{\text{Th}}$ is the Thomson cross section, which signifies that the scattering is that of electromagnetic waves from electrons. The physics of the system under study is buried in the *dynamic structure factor* $S(q, E)$, which is a function of momentum transfer q and energy transfer E . The dynamic structure factor contains all non-resonant electronic excitations, including not only the core-electron excitations observed in XRS but also e.g. plasmons, the collective fluctuations of valence electrons, and Compton scattering.

Similarity to x-ray absorption

It was shown by Yukio Mizuno and Yoshihiro Ohmura in 1967 that at small momentum transfers q the XRS contribution of the dynamic structure factor is proportional to the x-ray absorption spectrum. The main difference is that while the polarization vector of light couples to momentum of the absorbing electron in XAS, in XRS the momentum of the incident photon couples to the charge of the electron. Because of this, the momentum transfer of XRS plays the role of photon polarization of XAS.

References

- [1] Schülke, W (2007). *Electron dynamics studied by inelastic x-ray scattering*. Oxford University Press.

External links

- Synchrotron radiation sources worldwide (<http://www.lightsources.org/>)

References

See also

Structure determination

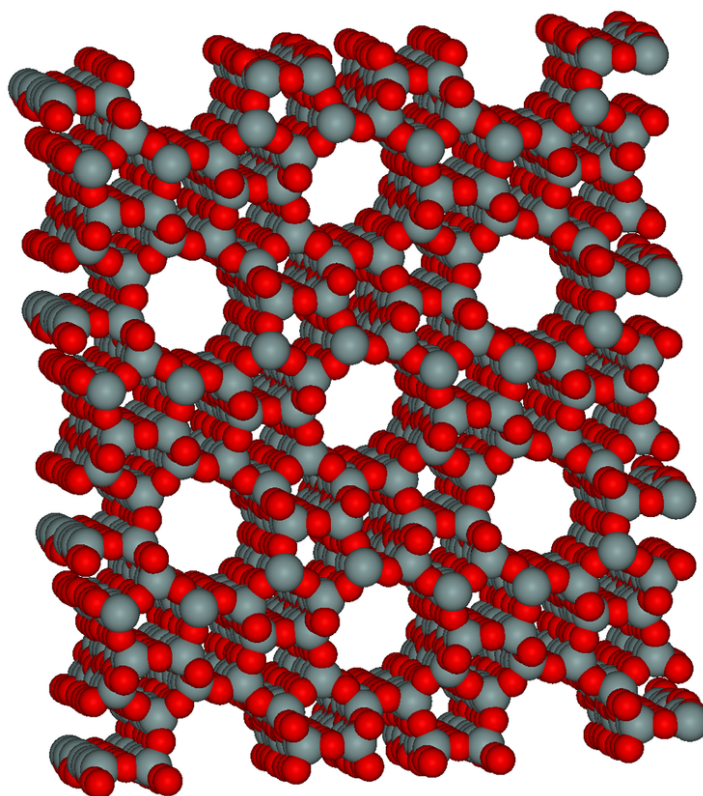
1. REDIRECT Chemical structure

X-ray crystallography

X-ray crystallography is a method of determining the arrangement of atoms within a crystal, in which a beam of X-rays strikes a crystal and scatters into many different directions. From the angles and intensities of these scattered beams, a crystallographer can produce a three-dimensional picture of the density of electrons within the crystal. From this electron density, the mean positions of the atoms in the crystal can be determined, as well as their chemical bonds, their disorder and various other information.

Since many materials can form crystals — such as salts, metals, minerals, semiconductors, as well as various inorganic, organic and biological molecules — X-ray crystallography has been

fundamental in the development of many scientific fields. In its first decades of use, this method determined the size of atoms, the lengths and types of chemical bonds, and the atomic-scale differences among various materials, especially minerals and alloys. The method also revealed the structure and functioning of many biological molecules, including vitamins, drugs, proteins and nucleic acids such as DNA. X-ray crystallography is still the chief method for characterizing the atomic structure of new materials and in discerning materials that appear similar by other experiments. X-ray crystal structures can also account for unusual electronic or elastic properties of a material, shed light on chemical interactions and processes, or serve as the basis for designing pharmaceuticals against



X-ray crystallography can locate every atom in a zeolite, an aluminosilicate with many important applications, such as water purification.

diseases.

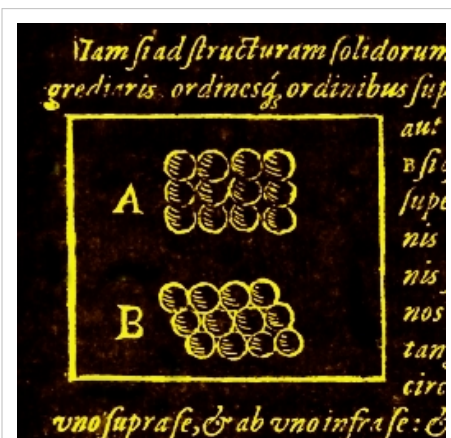
After a crystal has been obtained or grown in the laboratory, it is mounted on a goniometer and gradually rotated while being bombarded with X-rays, producing a diffraction pattern of regularly spaced spots known as *reflections*. The two-dimensional images taken at different rotations are converted into a three-dimensional model of the density of electrons within the crystal using the mathematical method of Fourier transforms, combined with chemical data known for the sample. Poor resolution (fuzziness) or even errors may result if the crystals are too small, or not uniform enough in their internal makeup.

X-ray crystallography is related to several other methods for determining atomic structures. Similar diffraction patterns can be produced by scattering electrons or neutrons, which are likewise interpreted as a Fourier transform. If single crystals of sufficient size cannot be obtained, various X-ray scattering methods can be applied to obtain less detailed information; such methods include fiber diffraction, powder diffraction and small-angle X-ray scattering (SAXS). In all these methods, the scattering is elastic; the scattered X-rays have the same wavelength as the incoming X-ray. By contrast, *inelastic* X-ray scattering methods are useful in studying excitations of the sample, rather than the distribution of its atoms.

History

Early scientific history of crystals and X-rays

Crystals have long been admired for their regularity and symmetry, but they were not investigated scientifically until the 17th century. Johannes Kepler hypothesized in his work *Strena seu de Nive Sexangula* (1611) that the hexagonal symmetry of snowflake crystals was due to a regular packing of spherical water particles.^[1]



Drawing of square (Figure A, above) and hexagonal (Figure B, below) packing from Kepler's work, *Strena seu de Nive Sexangula*.

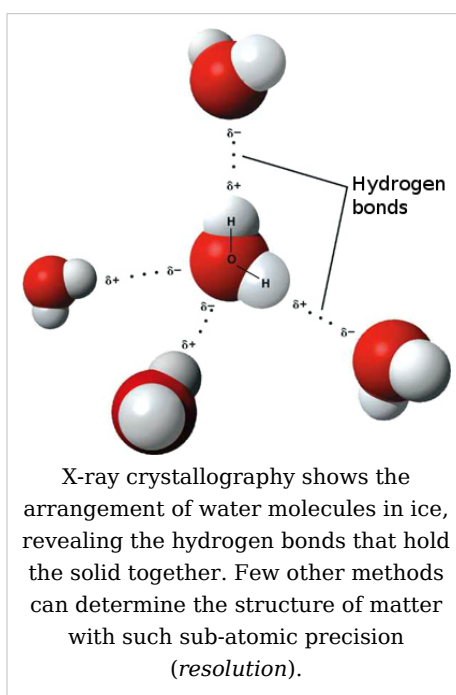


As shown by X-ray crystallography, the hexagonal symmetry of snowflakes results from the tetrahedral arrangement of hydrogen bonds about each water molecule. The water molecules are arranged similarly to the silicon atoms in the tridymite polymorph of SiO_2 . The resulting crystal structure has hexagonal symmetry when viewed along a principal axis.

Crystal symmetry was first investigated experimentally by Nicolas Steno (1669), who showed that the angles between the faces are the same in every exemplar of a particular type of crystal,^[2] and by René Just Haüy (1784), who discovered that every face of a crystal can be described by simple stacking patterns of blocks of the same shape and size. Hence, William Hallows Miller in 1839 was able to give each face a unique label of three small integers, the Miller indices which are still used today for identifying crystal faces. Haüy's study led to the correct idea that crystals are a regular three-dimensional array (a Bravais lattice) of atoms and molecules; a single unit cell is repeated indefinitely along three principal directions that are not necessarily perpendicular. In the 19th century, a complete catalog of the possible symmetries of a crystal was worked out by Johann Hessel,^[3] Auguste Bravais,^[4] Yevgraf Fyodorov,^[5] Arthur Schönflies^[6] and (belatedly) William Barlow. On the basis of the available data and physical reasoning, Barlow proposed several crystal structures in the 1880s that were validated later by

X-ray crystallography;^[7] however, the available data were too few in the 1880s to accept his models as conclusive.

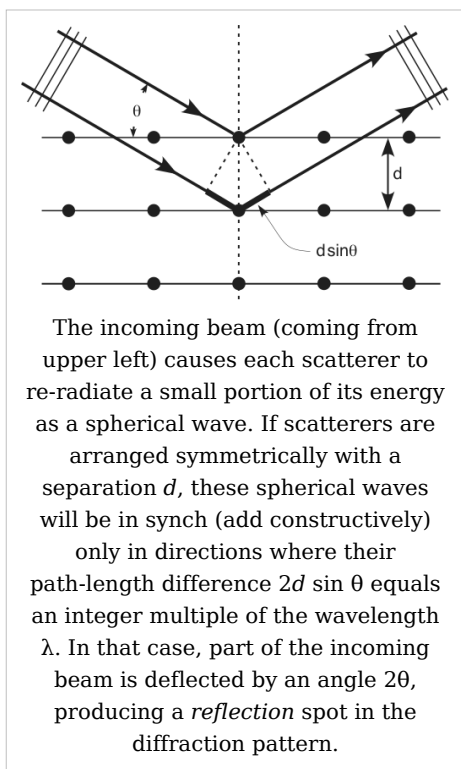
X-rays were discovered by Wilhelm Conrad Röntgen in 1895, just as the studies of crystal symmetry were being concluded. Physicists were initially uncertain of the nature of X-rays, although it was soon suspected (correctly) that they were waves of electromagnetic radiation, in other words, another form of light. At that time, the wave model of light — specifically, the Maxwell theory of electromagnetic radiation — was well accepted among scientists, and experiments by Charles Glover Barkla showed that X-rays exhibited phenomena associated with electromagnetic waves, including transverse polarization and spectral lines akin to those observed in the visible wavelengths. Single-slit experiments in the laboratory of Arnold Sommerfeld suggested the wavelength of X-rays was roughly 1 Angström, one ten millionth of a millimetre. However, X-rays are composed of photons, and thus are not only waves of electromagnetic radiation but also exhibit particle-like properties. The photon concept was introduced by Albert Einstein in 1905,^[8] but it was not broadly accepted until 1922,^[9] ^[10] when Arthur Compton confirmed it by the scattering of X-rays from electrons.^[11] Therefore, these particle-like properties of X-rays,



such as their ionization of gases, caused William Henry Bragg to argue in 1907 that X-rays were *not* electromagnetic radiation.^[12] Nevertheless, Bragg's view was not broadly

accepted and the observation of X-ray diffraction in 1912^[13] confirmed for most scientists that X-rays were a form of electromagnetic radiation.

X-ray analysis of crystals



Crystals are regular arrays of atoms, and X-rays can be considered waves of electromagnetic radiation. Atoms scatter X-ray waves, primarily through the atoms' electrons. Just as an ocean wave striking a lighthouse produces secondary circular waves emanating from the lighthouse, so an X-ray striking an electron produces secondary spherical waves emanating from the electron. This phenomenon is known as elastic scattering, and the electron (or lighthouse) is known as the *scatterer*. A regular array of scatterers produces a regular array of spherical waves. Although these waves cancel one another out in most directions through destructive interference, they add constructively in a few specific directions, determined by Bragg's law:

$$2d \sin \theta = n\lambda$$

where d is the spacing between diffracting planes, θ is the incident angle, n is any integer, and λ is the wavelength of the beam. These specific directions appear as spots on the diffraction pattern, often called *reflections*. Thus, X-ray diffraction results from an electromagnetic wave (the X-ray) impinging on a regular array of scatterers (the repeating arrangement of atoms within the crystal).

X-rays are used to produce the diffraction pattern because their wavelength λ is typically the same order of magnitude (1-100 Ångströms) as the spacing d between planes in the crystal. In principle, any wave impinging on a regular array of scatterers produces diffraction, as predicted first by Francesco Maria Grimaldi in 1665. To produce significant diffraction, the spacing between the scatterers and the wavelength of the impinging wave should be roughly similar in size. For illustration, the diffraction of sunlight through a bird's feather was first reported by James Gregory in the later 17th century. The first man-made diffraction gratings for visible light were constructed by David Rittenhouse in 1787, and Joseph von Fraunhofer in 1821. However, visible light has too long a wavelength (typically, 5500 Ångströms) to observe diffraction from crystals. However, prior to the first X-ray diffraction experiments, the spacings between lattice planes in a crystal were not known with certainty.

The idea that crystals could be used as a diffraction grating for X-rays arose in 1912 in a conversation between Paul Peter Ewald and Max von Laue in the English Garden in Munich. Ewald had proposed a resonator model of crystals for his thesis, but this model could not be validated using visible light, since the wavelength was much larger than the

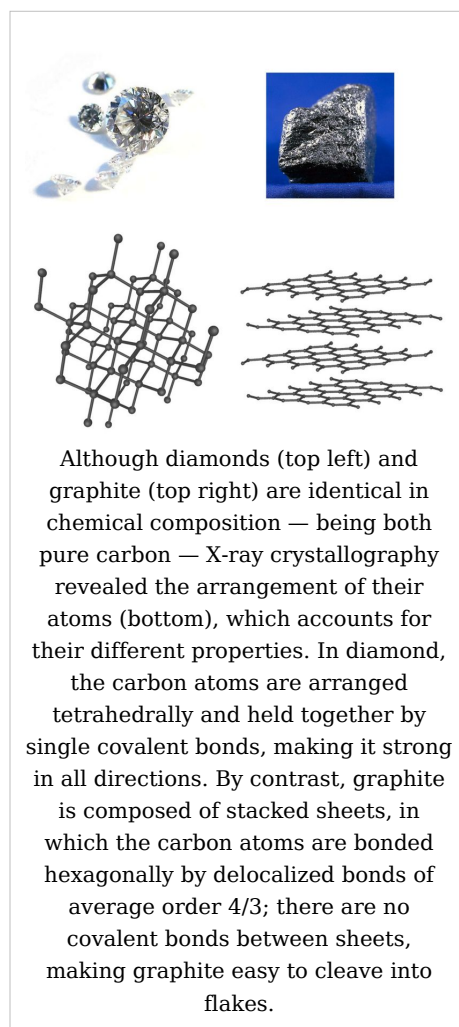
spacing between the resonators. Von Laue realized that electromagnetic radiation of a shorter wavelength was needed to observe such small spacings, and suggested that X-rays might have a wavelength comparable to the unit-cell spacing in crystals. Von Laue worked with two technicians, Walter Friedrich and his assistant Paul Knipping, to shine a beam of X-rays through a copper sulphate crystal and record its diffraction on a photographic plate. After being developed, the plate showed a large number of well-defined spots arranged in a pattern of intersecting circles around the spot produced by the central beam.^[13] ^[14] Von Laue developed a law that connects the scattering angles and the size and orientation of the unit-cell spacings in the crystal, for which he was awarded the Nobel Prize in Physics in 1914.^[15]

As described in the mathematical derivation below, the X-ray scattering is determined by the density of electrons within the crystal. Since the energy of an X-ray is much greater than that of an atomic electron, the scattering may be modeled as Thomson scattering, the interaction of an electromagnetic ray with a free electron. This model is generally adopted to describe the polarization of the scattered radiation. The intensity of Thomson scattering declines as $1/m^2$ with the mass m of the charged particle that is scattering the radiation; hence, the atomic nuclei, which are thousands of times heavier than an electron, contribute negligibly to the scattered X-rays.

Development from 1912 to 1920

After Von Laue's pioneering research, the field developed rapidly, most notably by physicists William Lawrence Bragg and his father William Henry Bragg. In 1912-1913, the younger Bragg developed Bragg's law, which connects the observed scattering with reflections from evenly spaced planes within the crystal.^[16] The earliest structures were generally simple and marked by one-dimensional symmetry. However, as computational and experimental methods improved over the next decades, it became feasible to deduce reliable atomic positions for more complicated two- and three-dimensional arrangements of atoms in the unit-cell.

The potential of X-ray crystallography for determining the structure of molecules and minerals — then only known vaguely from chemical and hydrodynamic experiments — was realized immediately. The earliest structures were simple inorganic crystals and minerals, but even these revealed fundamental laws of physics and chemistry. The first atomic-resolution structure to be solved (in 1914) was that of table salt.^[17] (When an atomic structure is determined by X-ray crystallography, it is said to be "solved".) The distribution of electrons in the table-salt structure



showed that crystals are not necessarily comprised of covalently bonded molecules, and proved the existence of ionic compounds.^[18] The structure of diamond was solved in the same year,^[19] proving the tetrahedral arrangement of its chemical bonds and showing that the C-C single bond was 1.52 Å. Other early structures included copper,^[20] calcium fluoride (CaF_2 , also known as *fluorite*), calcite (CaCO_3) and pyrite (FeS_2)^[21] in 1914; spinel (MgAl_2O_4) in 1915;^[22] the rutile and anatase forms of titanium dioxide (TiO_2) in 1916;^[23] pyrochroite and, by extension, brucite [$\text{Mn}(\text{OH})_2$ and $\text{Mg}(\text{OH})_2$, respectively] in 1919;^[24] and wurtzite (hexagonal ZnS) in 1920.^[25]

The structure of graphite was solved in 1916^[26] by the related method of powder diffraction,^[27] which was developed by Peter Debye and Paul Scherrer and, independently, by Albert Hull in 1917.^[28] The structure of graphite was determined from single-crystal diffraction in 1924 by two groups independently.^[29] ^[30] Hull also used the powder method to determine the structures of various metals, such as iron^[31] and magnesium.^[32]

Contributions to chemistry and material science

X-ray crystallography has led to a better understanding of chemical bonds and non-covalent interactions. The initial studies revealed the typical radii of atoms, and confirmed many theoretical models of chemical bonding, such as the tetrahedral bonding of carbon in the diamond structure,^[19] the octahedral bonding of metals observed in ammonium hexachloroplatinate (IV),^[33] and the resonance observed in the planar carbonate group^[21] and in aromatic molecules.^[34] ^[35] Kathleen Lonsdale's 1928 structure of hexamethylbenzene^[35] established the hexagonal symmetry of benzene and showed a clear difference in bond length between the aliphatic C-C bonds and aromatic C-C bonds; this finding led to the idea of resonance between chemical bonds, which had profound consequences for the development of chemistry.^[36] Her conclusions were anticipated by William Henry Bragg, who published models of naphthalene and anthracene in 1921 based on other molecules, an early form of molecular replacement.^[34]

Also in the 1920s, Victor Moritz Goldschmidt and later Linus Pauling developed rules for eliminating chemically unlikely structures and for determining the relative sizes of atoms. These rules led to the structure of brookite (1928) and an understanding of the relative stability of the rutile, brookite and anatase forms of titanium oxide.

The distance between two covalently bonded atoms is a sensitive measure of the bond strength and its bond order; thus, X-ray crystallographic studies have led to the discovery of even more exotic types of bonding in inorganic chemistry, such as metal-metal double bonds,^[37] metal-metal quadruple bonds,^[38] and three-center, two-electron bonds.^[39] X-ray crystallography — or, strictly speaking, an inelastic Compton scattering experiment — has also provided evidence for the partially covalent character of hydrogen bonds.^[40] In the field of organometallic chemistry, the X-ray structure of ferrocene initiated scientific studies of sandwich compounds,^[41] while that of Zeise's salt stimulated research into "back bonding" and metal- π complexes in general.^[42] Finally, X-ray crystallography had a pioneering role in the development of supramolecular chemistry, particularly in clarifying the structures of the crown ethers and the principles of host-guest chemistry.

In material sciences, many complicated inorganic and organometallic systems have been analyzed using single-crystal methods, such as fullerenes, metalloporphyrins, and other complicated compounds. Single-crystal diffraction is also used in the pharmaceutical industry, due to recent problems with polymorphs. The major factors affecting the quality of

single-crystal structures are the crystal's size and regularity; recrystallization is a commonly used technique to improve these factors in small-molecule crystals. The Cambridge Structural Database contains over 400,000 structures; over 99% of these structures were determined by X-ray diffraction.

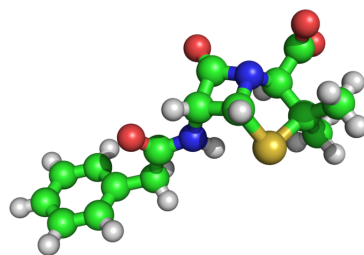
Mineralogy and metallurgy

Since the 1920s, X-ray diffraction has been the principal method for determining the arrangement of atoms in minerals and metals. The application of X-ray crystallography to mineralogy began with the structure of garnet, which was determined in 1924 by Menzer. A systematic X-ray crystallographic study of the silicates was undertaken in the 1920s. This study showed that, as the Si/O ratio is altered, the silicate crystals exhibit significant changes in their atomic arrangements. Machatschki extended these insights to minerals in which aluminium substitutes for the silicon atoms of the silicates. The first application of X-ray crystallography to metallurgy likewise occurred in the mid-1920s.^[43] Most notably, Linus Pauling's structure of the alloy Mg_2Sn ^[44] led to his theory of the stability and structure of complex ionic crystals.^[45]

Early organic and small biological molecules

The first structure of an organic compound, hexamethylenetetramine, was solved in 1923.^[46] This was followed by several studies of long-chain fatty acids, which are an important component of biological membranes.^[47] In the 1930s, the structures of much larger molecules with two-dimensional complexity began to be solved. A significant advance was the structure of phthalocyanine,^[48] a large planar molecule that is closely related to porphyrin molecules important in biology, such as heme, corrin and chlorophyll.

X-ray crystallography of biological molecules took off with Dorothy Crowfoot Hodgkin, who solved the structures of cholesterol (1937), vitamin B12 (1945) and penicillin (1954), for which she was awarded the Nobel Prize in Chemistry in 1964. In 1969, she succeeded in solving the structure of insulin, on which she worked for over thirty years.^[49]



The three-dimensional structure of penicillin, for which Dorothy Crowfoot Hodgkin was awarded the Nobel Prize in Chemistry in 1964. The green, white, red, yellow and blue spheres represent atoms of carbon, hydrogen, oxygen, sulfur and nitrogen, respectively.



Ribbon diagram of the structure of myoglobin, showing colored alpha helices. Such proteins are long, linear molecules with thousands of atoms; yet the relative position of each atom has been determined with sub-atomic resolution by X-ray crystallography. Since it is difficult to visualize all the atoms at once, the ribbon shows the rough path of the protein polymer from its N-terminus (blue) to its C-terminus (red).

Protein crystallography

Crystal structures of proteins (which are irregular and hundreds of times larger than cholesterol) began to be solved in the late 1950s, beginning with the structure of sperm whale myoglobin by Max Perutz and Sir John Cowdery Kendrew, for which they were awarded the Nobel Prize in Chemistry in 1962.^[50] Since that success, over 48970 X-ray crystal structures of proteins, nucleic acids and other biological molecules have been determined.^[51] For comparison, the nearest competing method, nuclear magnetic resonance (NMR) spectroscopy has produced 7806 structures.^[52] Moreover, crystallography can solve structures of arbitrarily large molecules, whereas solution-state NMR is restricted to relatively small molecules (less than 70 kDa). X-ray crystallography is now used routinely by scientists to determine how a pharmaceutical interacts with its protein target and what changes might be advisable to improve it.^[53] However, intrinsic membrane proteins remain challenging to crystallize because they require detergents or other means to solubilize them in isolation, and such detergents often interfere with crystallization. Such membrane proteins

are a large component of the genome and include many proteins of great physiological importance, such as ion channels and receptors.^{[54] [55]}

Relationship to other scattering techniques

Elastic vs. inelastic scattering

X-ray crystallography is a form of elastic scattering; the outgoing X-rays have the same energy as the incoming X-rays, only with altered direction. Since the energy of a photon is inversely proportional to its wavelength, elastic scattering means that the outgoing photons have the same wavelength as the incoming photons. By contrast, *inelastic scattering* occurs when energy is transferred from the incoming X-ray to the crystal, e.g., by exciting an inner-shell electron to a higher energy level. Such inelastic scattering changes the wavelength of the outgoing beam, making it longer and less energetic. Inelastic scattering is useful for probing such excitations of matter, but are not as useful in determining the distribution of scatterers within the matter, which is the goal of X-ray crystallography.

X-rays range in wavelength from 10 to 0.01 nanometers; a typical wavelength used for crystallography is roughly 1 Å (0.1 nm), which is on the scale of covalent chemical bonds and the radius of a single atom. Longer-wavelength photons (such as ultraviolet radiation) would not have sufficient resolution to determine the atomic positions. At the other extreme, shorter-wavelength photons such as gamma rays are difficult to produce in large numbers, difficult to focus, and interact too strongly with matter, producing

particle-antiparticle pairs. Therefore, X-rays are the "sweetspot" for wavelength when determining atomic-resolution structures from the scattering of electromagnetic radiation.

Other types of X-ray scattering

X-ray diffraction involves the scattering of X-rays from a single crystal. Other forms of elastic X-ray scattering include powder diffraction, SAXS and several types of X-ray fiber diffraction, which was used by Rosalind Franklin in determining the double-helix structure of DNA. In general, X-ray diffraction produces isolated spots ("reflections"), while the other methods produce smooth, continuous scattering. In general, X-ray diffraction offers more structural information than these other techniques; however, it requires a sufficiently large and regular crystal, which is not always possible to obtain.

All of these scattering methods generally use *monochromatic* X-rays, which are restricted to a single wavelength with minor deviations. A broad spectrum of X-rays (that is, a blend of X-rays with different wavelengths) can also be used to carry out X-ray diffraction, a technique known as the Laue method. This is the method used in the original discovery of X-ray diffraction. Laue scattering provides much structural information with only a short exposure to the X-ray beam, and is therefore used in structural studies of very rapid events (time-resolved X-ray crystallography). However, it is not as well-suited as monochromatic scattering for determining the full atomic structure of a crystal. It is better suited to crystals with relatively simple atomic arrangements, such as minerals.

The Laue back reflection mode records X-rays scattered backwards also from a broad spectrum source. This is useful if the sample is too thick or bulky for X-rays to transmit through it. The diffracting planes in the crystal are determined by knowing that the normal to the diffracting plane bisects the angle between the incident beam and the diffracted beam. A Greninger chart can be used ^[56] to interpret the back reflection Laue photograph. The X-calibre RTXDB and MWL 110 are commercial systems for Laue back reflection pattern recording. This technique can be used in materials analysis or nondestructive testing.

Electron and neutron diffraction

Other particles, such as electrons and neutrons, may be used to produce a diffraction pattern. Although electron, neutron, and X-ray scattering use very different equipment, the resulting diffraction patterns are analyzed using the same coherent diffraction imaging techniques.

As derived below, the electron density within the crystal and the diffraction patterns are related by a simple mathematical method, the Fourier transform, which allows the density to be calculated relatively easily from the patterns. However, this works only if the scattering is *weak*, i.e., if the scattered beams are much less intense than the incoming beam. Weakly scattered beams pass through the remainder of the crystal without undergoing a second scattering event. Such re-scattered waves are called "secondary scattering" and hinder the calculation of the density of scatterers. Any sufficiently thick crystal will produce secondary scattering but since X-rays interact relatively weakly with the electrons, this is generally not a significant concern. By contrast, electron beams may produce strong secondary scattering even for very small crystals (e.g., 100 μm) used in X-ray crystallography. In such cases, extremely thin samples, roughly 100 nanometers or less, must be used to avoid secondary scattering; the primary scattered electron beams

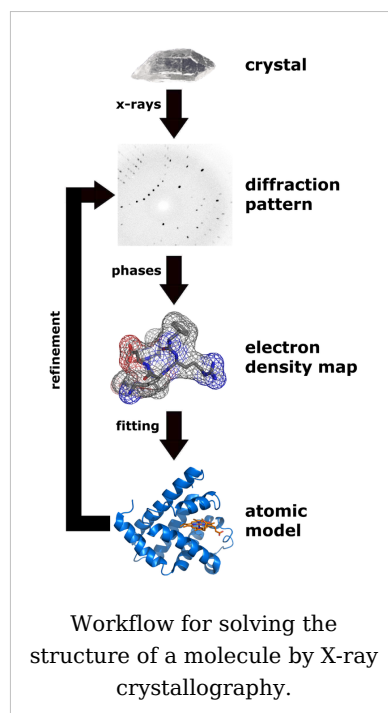
leave the sample before they have a chance to undergo secondary scattering. Since this thickness corresponds roughly to the diameter of many viruses, a promising direction is the electron diffraction of isolated macromolecular assemblies, such as viral capsids and molecular machines, which may be carried out with a cryo-electron microscope.

Neutron diffraction is an excellent method for structure determination, although it has been difficult to obtain intense, monochromatic beams of neutrons in sufficient quantities. Traditionally, nuclear reactors have been used, although the new Spallation Neutron Source holds much promise in the near future. Being uncharged, neutrons scatter much more readily from the atomic nuclei rather than from the electrons. Therefore, neutron scattering is very useful for observing the positions of light atoms with few electrons, especially hydrogen, which is essentially invisible in the X-ray diffraction of larger molecules. Neutron scattering also has the remarkable property that the solvent can be made invisible by adjusting the ratio of normal water, H_2O , and heavy water, D_2O .

Methods

Overview of single-crystal X-ray diffraction

The oldest and most precise method of X-ray crystallography is *single-crystal X-ray diffraction*, in which a beam of X-rays strikes a single crystal, producing scattered beams. When they land on a piece of film or other detector, these beams make a *diffraction pattern* of spots; the strengths and angles of these beams are recorded as the crystal is gradually rotated.^[57] Each spot is called a *reflection*, since it corresponds to the reflection of the X-rays from one set of evenly spaced planes within the crystal. For single crystals of sufficient purity and regularity, X-ray diffraction data can determine the mean chemical bond lengths and angles to within a few thousandths of an Ångström and to within a few tenths of a degree, respectively. The atoms in a crystal are also not static, but oscillate about their mean positions, usually by less than a few tenths of an Ångström. X-ray crystallography allows the size of these oscillations to be measured quantitatively.



Procedure

The technique of single-crystal X-ray crystallography has three basic steps. The first — and often most difficult — step is to obtain an adequate crystal of the material under study. The crystal should be sufficiently large (typically larger than 100 micrometres in all dimensions), pure in composition and regular in structure, with no significant internal imperfections such as cracks or twinning. A small or irregular crystal will give fewer and less reliable data, from which it may be impossible to determine the atomic arrangement.

In the second step, the crystal is placed in an intense beam of X-rays, usually of a single wavelength (*monochromatic X-rays*), producing the regular pattern of reflections. As the

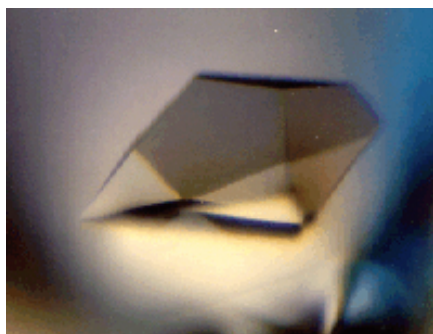
crystal is gradually rotated, previous reflections disappear and new ones appear; the intensity of every spot is recorded at every orientation of the crystal. Multiple data sets may have to be collected, with each set covering slightly more than half a full rotation of the crystal and typically containing tens of thousands of reflection intensities.

In the third step, these data are combined computationally with complementary chemical information to produce and refine a model of the arrangement of atoms within the crystal. The final, refined model of the atomic arrangement — now called a *crystal structure* — is usually stored in a public database.

Limitations

As the crystal's repeating unit, its unit cell, becomes larger and more complex, the atomic-level picture provided by X-ray crystallography becomes less well-resolved (more "fuzzy") for a given number of observed reflections. Two limiting cases of X-ray crystallography—"small-molecule" and "macromolecular" crystallography—are often discerned. *Small-molecule crystallography* typically involves crystals with fewer than 100 atoms in their asymmetric unit; such crystal structures are usually so well resolved that the atoms can be discerned as isolated "blobs" of electron density. By contrast, *macromolecular crystallography* often involves tens of thousands of atoms in the unit cell. Such crystal structures are generally less well-resolved (more "smeared out"); the atoms and chemical bonds appear as tubes of electron density, rather than as isolated atoms. In general, small molecules are also easier to crystallize than macromolecules; however, X-ray crystallography has proven possible even for viruses with hundreds of thousands of atoms.

Crystallization



A protein crystal seen under a microscope. Crystals used in X-ray crystallography are generally smaller than a millimeter across.

Although crystallography can be used to characterize the disorder in an impure or irregular crystal, crystallography generally requires a pure crystal of high regularity to solve for the structure of a complicated arrangement of atoms. Pure, regular crystals can sometimes be obtained from natural or man-made materials, such as samples of metals, minerals or other macroscopic materials. The regularity of such crystals can sometimes be improved with annealing and other methods. However, in many cases, obtaining a diffraction-quality crystal is the chief barrier to solving its atomic-resolution structure.^[58]

Small-molecule and macromolecular crystallography differ in the range of possible techniques used to produce diffraction-quality crystals. Small molecules generally have few degrees of conformational freedom, and may be crystallized by a wide range of methods, such as chemical vapor deposition and recrystallisation. By contrast, macromolecules generally have many degrees of freedom and their crystallization must be carried out to maintain a stable structure. For example, proteins and larger RNA molecules cannot be crystallized if their tertiary structure has been unfolded; therefore, the range of crystallization conditions is restricted to solution conditions in which such molecules remain folded.

Protein crystals are almost always grown in solution. The most common approach is to lower the solubility of its component molecules very gradually; however, if this is done too quickly, the molecules will precipitate from solution, forming a useless dust or amorphous gel on the bottom of the container. Crystal growth in solution is characterized by two steps: *nucleation* of a microscopic crystallite (possibly having only 100 molecules), followed by *growth* of that crystallite, ideally to a diffraction-quality crystal.^[59] The solution conditions that favor the first step (nucleation) are not always the same conditions that favor the second step (its subsequent growth). The crystallographer's goal is to identify solution conditions that favor the development of a single, large crystal, since larger crystals offer improved resolution of the molecule. Consequently, the solution conditions should *disfavor* the first step (nucleation) but *favor* the second (growth), so that only one large crystal forms per droplet. If nucleation is favored too much, a shower of small crystallites will form in the droplet, rather than one large crystal; if favored too little, no crystal will form whatsoever.

It is extremely difficult to predict good conditions for nucleation or growth of well-ordered crystals.^[60] In practice, favorable conditions are identified by *screening*; a very large batch of the molecules is prepared, and a wide variety of crystallization solutions are tested.^[61] Hundreds, even thousands, of solution conditions are generally tried before finding one that succeeds in crystallizing the molecules. The various conditions can use one or more physical mechanisms to lower the solubility of the molecule; for example, some may change the pH, some contain salts of the Hofmeister series or chemicals that lower the dielectric constant of the solution, and still others contain large polymers such as polyethylene glycol that drive the molecule out of solution by entropic effects. It is also common to try several temperatures for encouraging crystallization, or to gradually lower the temperature so that the solution becomes supersaturated. These methods require large amounts of the target molecule, as they use high concentration of the molecule(s) to be crystallized. Due to the difficulty in obtaining such large quantities (milligrams) of crystallisation grade protein, dispensing robots have been developed that are capable of accurately dispensing crystallisation trial drops that are of the order on 100 nanoliters in volume. This means that roughly 10-fold less protein is used per-experiment when compared to crystallisation trials setup by hand (on the order on 1 microliter).^[62]

Several factors are known to inhibit or mar crystallization. The growing crystals are generally held at a constant temperature and protected from shocks or vibrations that might disturb their crystallization. Impurities in the molecules or in the crystallization solutions are often inimical to crystallization. Conformational flexibility in the molecule also tends to make crystallization less likely, due to entropy. Ironically, molecules that tend to self-assemble into regular helices are often unwilling to assemble into crystals. Crystals can be marred by twinning, which can occur when a unit cell can pack equally favorably in multiple orientations; although recent advances in computational methods have begun to allow the structures of twinned crystals to be solved, it is still very difficult. Having failed to crystallize a target molecule, a crystallographer may try again with a slightly modified version of the molecule; even small changes in molecular properties can lead to large differences in crystallization behavior.

Data collection

Mounting the crystal

Once they are full-grown, the crystals are mounted so that they may be held in the X-ray beam and rotated. There are several methods of mounting. Although crystals were once loaded into glass capillaries with the crystallization solution (the mother liquor), a more modern approach is to scoop the crystal up in a tiny loop, made of nylon or plastic and attached to a solid rod, that is then flash-frozen with liquid nitrogen.^[63] This freezing reduces the radiation damage of the X-rays, as well as the noise in the Bragg peaks due to thermal motion (the Debye-Waller effect). However, untreated crystals often crack if flash-frozen; therefore, they are generally pre-soaked in a cryoprotectant solution before freezing.^[64] Unfortunately, this pre-soak may itself cause the crystal to crack, ruining it for crystallography. Generally, successful cryo-conditions are identified by trial and error.

The capillary or loop is mounted on a goniometer, which allows it to be positioned accurately within the X-ray beam and rotated. Since both the crystal and the beam are often very small, the crystal must be centered within the beam to within roughly 25 micrometres accuracy, which is aided by a camera focused on the crystal. The most common type of goniometer is the "kappa goniometer", which offers three angles of rotation: the ω angle, which rotates about an axis roughly perpendicular to the beam; the κ angle, about an axis at roughly 50° to the ω axis; and, finally, the φ angle about the loop/capillary axis. When the κ angle is zero, the ω and φ axes are aligned. The κ rotation allows for convenient mounting of the crystal, since the arm in which the crystal is mounted may be swung out towards the crystallographer. The oscillations carried out during data collection (mentioned below) involve the ω axis only. An older type of goniometer is the four-circle goniometer, and its relatives such as the six-circle goniometer.

X-ray sources

The mounted crystal is then irradiated with a beam of monochromatic X-rays. The brightest and most useful X-ray sources are synchrotrons; their much higher luminosity allows for better resolution. They also make it convenient to tune the wavelength of the radiation, which is useful for multi-wavelength anomalous dispersion (MAD) phasing, described below. Synchrotrons are generally national facilities, each with several dedicated beamlines where data is collected around the clock, seven days a week.

Smaller, weaker X-ray sources are often used in laboratories to check the quality of crystals before bringing them to a synchrotron and sometimes to solve a crystal structure. In such systems, electrons are boiled off of a cathode and accelerated through a strong electric potential of roughly 50 kV; having reached a high speed, the electrons collide with a metal plate, emitting *bremsstrahlung* and some strong spectral lines corresponding to the excitation of inner-shell electrons of the metal. The most common metal used is copper, which can be kept cool easily, due to its high thermal conductivity, and which produces strong K_α and K_β lines. The K_β line is



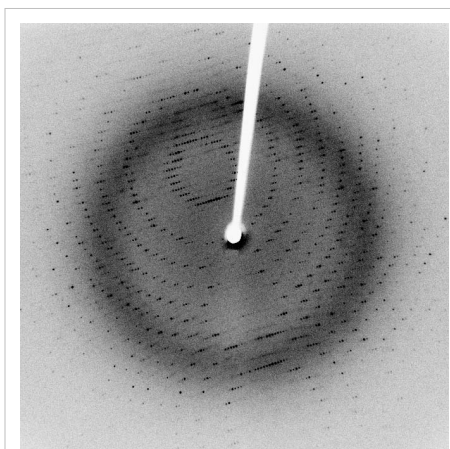
A diffractometer

sometimes suppressed with a thin layer (0.0005 in. thick) of nickel foil. The simplest and cheapest variety of sealed X-ray tube has a stationary anode (the Crookes tube) and produces *circa* 2 kW of X-ray radiation. The more expensive variety has a rotating-anode type source that produces *circa* 14 kW of X-ray radiation.

X-rays are generally filtered (by use of X-Ray Filters) to a single wavelength (made monochromatic) and collimated to a single direction before they are allowed to strike the crystal. The filtering not only simplifies the data analysis, but also removes radiation that degrades the crystal without contributing useful information. Collimation is done either with a collimator (basically, a long tube) or with a clever arrangement of gently curved mirrors. Mirror systems are preferred for small crystals (under 0.3 mm) or with large unit cells (over 150 Å).

Recording the reflections

When a crystal is mounted and exposed to an intense beam of X-rays, it scatters the X-rays into a pattern of spots or *reflections* that can be observed on a screen behind the crystal. A similar pattern may be seen by shining a laser pointer at a compact disc. The relative intensities of these spots provide the information to determine the arrangement of molecules within the crystal in atomic detail. The intensities of these reflections may be recorded with photographic film, an area detector or with a **charge-coupled device (CCD)** image sensor. The peaks at small angles correspond to low-resolution data, whereas those at high angles represent high-resolution data; thus, an upper limit on the eventual resolution of the structure can be determined from the first few images. Some measures of diffraction quality can be determined at this point, such as the mosaicity of the crystal and its overall disorder, as observed in the peak widths. Some pathologies of the crystal that would render it unfit for solving the structure can also be diagnosed quickly at this point.



An X-ray diffraction pattern of a crystallized enzyme. The pattern of spots (called *reflections*) can be used to determine the structure of the enzyme.

One image of spots is insufficient to reconstruct the whole crystal; it represents only a small slice of the full Fourier transform. To collect all the necessary information, the crystal must be rotated step-by-step through 180°, with an image recorded at every step; actually, slightly more than 180° is required to cover reciprocal space, due to the curvature of the Ewald sphere. However, if the crystal has a higher symmetry, a smaller angle such as 90° or 45° may be recorded. The axis of the rotation should generally be changed at least once, to avoid developing a "blind spot" in reciprocal space close to the rotation axis. It is customary to rock the crystal slightly (by 0.5-2°) to catch a broader region of reciprocal space.

Multiple data sets may be necessary for certain phasing methods. For example, MAD phasing requires that the scattering be recorded at least three (and usually four, for redundancy) wavelengths of the incoming X-ray radiation. A single crystal may degrade too much during the collection of one data set, owing to radiation damage; in such cases, data sets on multiple crystals must be taken.^[65]

Data analysis

Crystal symmetry, unit cell, and image scaling

Having recorded a series of diffraction patterns from the crystal, each corresponding to a different crystal orientation, the crystallographer must now convert these two-dimensional images into a three-dimensional model of the density of electrons throughout the crystal using the mathematical technique of Fourier transforms. (The relevance of this technique is explained below.) Roughly speaking, each spot corresponds to a different type of variation in the electron density; the crystallographer must determine *which* variation corresponds to *which* spot (*indexing*), the relative strengths of the spots in different images (*merging and scaling*) and how the variations should be combined to yield the total electron density (*phasing*).

In order to process the data, a crystallographer must first *index* the reflections within the multiple images recorded. This means identifying the dimensions of the unit cell and which image peak corresponds to which position in reciprocal space. A byproduct of indexing is to determine the symmetry of the crystal, i.e., its *space group*. Some space groups can be eliminated from the beginning, since they require symmetries known to be absent in the molecule itself. For example, reflection symmetries cannot be observed in chiral molecules; thus, only 65 space groups of 243 possible are allowed for protein molecules which are almost always chiral. Indexing is generally accomplished using an *autoindexing* routine.^[66] Having assigned symmetry, the data is then *integrated*. This converts the hundreds of images containing the thousands of reflections into a single file, consisting of (at the very least) records of the Miller index of each reflection, and an intensity for each reflection (at this state the file often also includes error estimates and measures of partiality (what part of a given reflection was recorded on that image)).

A full data set may consist of hundreds of separate images taken at different orientations of the crystal. The first step is to merge and scale these various images, that is, to identify which peaks appear in two or more images (*merging*) and to scale the relative images so that they have a consistent intensity scale. Optimizing the intensity scale is critical because the relative intensity of the peaks is the key information from which the structure is determined. The repetitive technique of crystallographic data collection and the often high symmetry of crystalline materials cause the diffractometer to record many symmetry-equivalent reflections multiple times. This allows a merging or symmetry related R-factor to be calculated based upon how similar are the measured intensities of symmetry equivalent reflections, thus giving a score to assess the quality of the data.

Initial phasing

The data collected from a diffraction experiment is a reciprocal space representation of the crystal lattice. The position of each diffraction 'spot' is governed by the size and shape of the unit cell, and the inherent symmetry within the crystal. The intensity of each diffraction 'spot' is recorded, and this intensity is proportional to the square of the *structure factor* amplitude. The structure factor is a complex number containing information relating to both the amplitude and phase of a wave. In order to obtain an interpretable *electron density map*, both amplitude and phase must be known (an electron density map allows a crystallographer to build a starting model of the molecule). The phase cannot be directly recorded during a diffraction experiment: this is known as the phase problem. Initial phase estimates can be obtained in a variety of ways:

- **Ab initio phasing**, aka **direct methods** - This is usually the method of choice for small molecules (<1000 non-hydrogen atoms), and has been used successfully to solve the phase problems for small proteins. If the resolution of the data is better than 1.4 Å (140 pm), direct methods can be used to obtain phase information, by exploiting known phase relationships between certain groups of reflections.^{[67] [68]}
- **Molecular replacement** - if a structure exists of a related structure, it can be used as a search model in molecular replacement to determine the orientation and position of the molecules within the unit cell. The phases obtained this way can be used to generate *electron density maps*.^[69]
- **Anomalous X-ray scattering** (*MAD or SAD phasing*) - the X-ray wavelength may be scanned past an absorption edge of an atom, which changes the scattering in a known way. By recording full sets of reflections at three different wavelengths (far below, far above and in the middle of the absorption edge) one can solve for the substructure of the anomalously diffracting atoms and thence the structure of the whole molecule. The most popular method of incorporating anomalous scattering atoms into proteins is to express the protein in a methionine auxotroph (a host incapable of synthesising methionine) in a media rich in Seleno-methionine, which contains Selenium atoms. A MAD experiment can then be conducted around the absorption edge, which should then yield the position of any methionine residues within the protein, providing initial phases.^[70]
- **Heavy atom methods** (ie MIR) - If electron-dense metal atoms can be introduced into the crystal, direct methods or Patterson-space methods can be used to determine their location and to obtain initial phases. Typically, a crystallographer can introduce such heavy atoms either by soaking the crystal in a heavy atom-containing solution, or by co-crystallization (growing the crystals in the presence of a heavy atom). As in MAD phasing, the changes in the scattering amplitudes can be interpreted to yield the phases. Although this is the original method by which protein crystal structures were solved, it has largely been superseded by MAD phasing with selenomethionine.^[69]

While all four of the above methods are used to solve the phase problem for protein crystallography, small molecule crystallography generally yields data suitable for structure solution using direct methods (*ab initio* phasing).

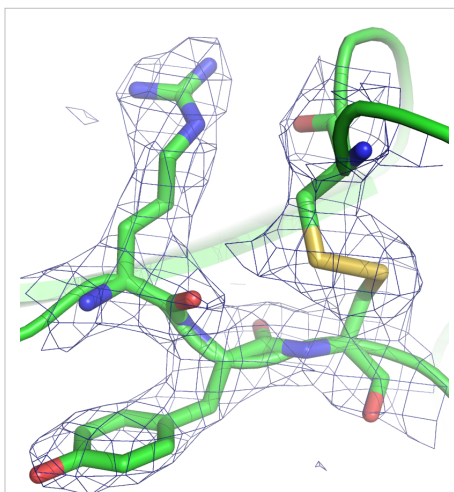
Model building and phase refinement

Having obtained initial phases, an initial model can be built. This model can be used to refine the phases, leading to an improved model, and so on. Given a model of some atomic positions, these positions and their respective Debye-Waller factors (accounting for the thermal motion of the atom - aka **B**-factors) can be refined to fit the observed diffraction data, ideally yielding a better set of phases. A new model can then be fit to the new electron density map and a further round of refinement is carried out. This continues until the correlation between the diffraction data and the model is maximized. The agreement is measured by an *R*-factor defined as

$$R = \frac{\sum_{\text{all reflections}} |F_o - F_c|}{\sum_{\text{all reflections}} |F_o|}$$

A similar quality criterion is R_{free} , which is calculated from a subset (~10%) of reflections that were not included in the structure refinement. Both *R* factors depend on the resolution of the data. As a rule of thumb, R_{free} should be approximately the resolution in Ångströms divided by 10; thus, a data-set with 2 Å resolution should yield a final R_{free} of roughly 0.2. Chemical bonding features such as stereochemistry, hydrogen bonding and distribution of bond lengths and angles are complementary measures of the model quality. Phase bias is a serious problem in such iterative model building. *Omit maps* are a common technique used to check for this.

It may not be possible to observe every atom of the crystallized molecule - it must be remembered that the resulting electron density is an average of all the molecules within the crystal. In some cases, there is too much residual disorder in those atoms, and the resulting electron density for atoms existing in many conformations is smeared to such an extent that it is no longer detectable in the electron density map. Weakly scattering atoms such as hydrogen are routinely invisible. It is also possible for a single atom to appear multiple times in an electron density map, e.g., if a protein sidechain has multiple (<4) allowed conformations. In still other cases, the crystallographer may detect that the covalent structure deduced for the molecule was incorrect, or changed. For example, proteins may be cleaved or undergo post-translational modifications that were not detected prior to the crystallization.



A protein crystal structure at 2.7 Å resolution. The mesh encloses the region in which the electron density exceeds a given threshold. The straight segments represent chemical bonds between the non-hydrogen atoms of an arginine (upper left), a tyrosine (lower left), a disulfide bond (upper right, in yellow), and some peptide groups (running left-right in the middle). The two curved green tubes represent spline fits to the polypeptide backbone.

Deposition of the structure

Once the model of a molecule's structure has been finalized, it is often deposited in a crystallographic database such as the Protein Data Bank (for protein structures) or the Cambridge Structural Database (for small molecules). Many structures obtained in private commercial ventures to crystallize medically relevant proteins, are not deposited in public crystallographic databases.

Diffraction theory

The main goal of X-ray crystallography is to determine the density of electrons $f(\mathbf{r})$ throughout the crystal, where \mathbf{r} represents the three-dimensional position vector within the crystal. To do this, X-ray scattering is used to collect data about its Fourier transform $F(\mathbf{q})$, which is inverted mathematically to obtain the density defined in real space, using the formula

$$f(\mathbf{r}) = \int \frac{d\mathbf{q}}{(2\pi)^3} F(\mathbf{q}) e^{i\mathbf{q}\cdot\mathbf{r}}$$

where the integral is summed over all possible values of \mathbf{q} . The three-dimensional real vector \mathbf{q} represents a point in reciprocal space, that is, to a particular oscillation in the electron density as one moves in the direction in which \mathbf{q} points. The length of \mathbf{q} corresponds to 2π divided by the wavelength of the oscillation. The corresponding formula for a Fourier transform will be used below

$$F(\mathbf{q}) = \int d\mathbf{r} f(\mathbf{r}) e^{-i\mathbf{q}\cdot\mathbf{r}}$$

where the integral is summed over all possible values of the position vector \mathbf{r} within the crystal.

The Fourier transform $F(\mathbf{q})$ is generally a complex number, and therefore has a magnitude $|F(\mathbf{q})|$ and a phase $\phi(\mathbf{q})$ related by the equation

$$F(\mathbf{q}) = |F(\mathbf{q})| e^{i\phi(\mathbf{q})}$$

The intensities of the reflections observed in X-ray diffraction give us the magnitudes $|F(\mathbf{q})|$ but not the phases $\phi(\mathbf{q})$. To obtain the phases, full sets of reflections are collected with known alterations to the scattering, either by modulating the wavelength past a certain absorption edge or by adding strongly scattering (i.e., electron-dense) metal atoms such as mercury. Combining the magnitudes and phases yields the full Fourier transform $F(\mathbf{q})$, which may be inverted to obtain the electron density $f(\mathbf{r})$.

Crystals are often idealized as being *perfectly* periodic. In that ideal case, the atoms are positioned on a perfect lattice, the electron density is perfectly periodic, and the Fourier transform $F(\mathbf{q})$ is zero except when \mathbf{q} belongs to the reciprocal lattice (the so-called *Bragg peaks*). In reality, however, crystals are not perfectly periodic; atoms vibrate about their mean position, and there may be disorder of various types, such as mosaicity, dislocations, various point defects, and heterogeneity in the conformation of crystallized molecules. Therefore, the Bragg peaks have a finite width and there may be significant *diffuse scattering*, a continuum of scattered X-rays that fall between the Bragg peaks.

Intuitive understanding by Bragg's law

An intuitive understanding of X-ray diffraction can be obtained from the Bragg model of diffraction. In this model, a given reflection is associated with a set of evenly spaced sheets running through the crystal, usually passing through the centers of the atoms of the crystal lattice. The orientation of a particular set of sheets is identified by its three Miller indices (h, k, l), and let their spacing be noted by d . William Lawrence Bragg proposed a model in which the incoming X-rays are scattered specularly (mirror-like) from each plane; from that assumption, X-rays scattered from adjacent planes will combine constructively (constructive interference) when the angle θ between the plane and the X-ray results in a path-length difference that is an integer multiple n of the X-ray wavelength λ .

$$2d \sin \theta = n\lambda$$

A reflection is said to be *indexed* when its Miller indices (or, more correctly, its reciprocal lattice vector components) have been identified from the known wavelength and the scattering angle 2θ . Such indexing gives the unit-cell parameters, the lengths and angles of the unit-cell, as well as its space group. Since Bragg's law does not interpret the relative intensities of the reflections, however, it is generally inadequate to solve for the arrangement of atoms within the unit-cell; for that, a Fourier transform method must be carried out.

Scattering as a Fourier transform

The incoming X-ray beam has a polarization and should be represented as a vector wave; however, for simplicity, let it be represented here as a scalar wave. We also ignore the complication of the time dependence of the wave and just focus on the wave's spatial dependence. Plane waves can be represented by a wave vector \mathbf{k}_{in} , and so the strength of the incoming wave at time $t=0$ is given by

$$Ae^{i\mathbf{k}_{\text{in}} \cdot \mathbf{r}}$$

At position \mathbf{r} within the sample, let there be a density of scatterers $f(\mathbf{r})$; these scatterers should produce a scattered spherical wave of amplitude proportional to the local amplitude of the incoming wave times the number of scatterers in a small volume dV about \mathbf{r}

$$\text{amplitude of scattered wave} = Ae^{i\mathbf{k} \cdot \mathbf{r}} S f(\mathbf{r}) dV$$

where S is the proportionality constant.

Let's consider the fraction of scattered waves that leave with an outgoing wave-vector of \mathbf{k}_{out} and strike the screen at $\mathbf{r}_{\text{screen}}$. Since no energy is lost (elastic, not inelastic scattering), the wavelengths are the same as are the magnitudes of the wave-vectors $|\mathbf{k}_{\text{in}}| = |\mathbf{k}_{\text{out}}|$. From the time that the photon is scattered at \mathbf{r} until it is absorbed at $\mathbf{r}_{\text{screen}}$, the photon undergoes a change in phase

$$e^{i\mathbf{k}_{\text{out}} \cdot (\mathbf{r}_{\text{screen}} - \mathbf{r})}$$

The net radiation arriving at $\mathbf{r}_{\text{screen}}$ is the sum of all the scattered waves throughout the crystal

$$AS \int d\mathbf{r} f(\mathbf{r}) e^{i\mathbf{k}_{\text{in}} \cdot \mathbf{r}} e^{i\mathbf{k}_{\text{out}} \cdot (\mathbf{r}_{\text{screen}} - \mathbf{r})} = AS e^{i\mathbf{k}_{\text{out}} \cdot \mathbf{r}_{\text{screen}}} \int d\mathbf{r} f(\mathbf{r}) e^{i(\mathbf{k}_{\text{in}} - \mathbf{k}_{\text{out}}) \cdot \mathbf{r}}$$

which may be written as a Fourier transform

$$AS e^{i\mathbf{k}_{\text{out}} \cdot \mathbf{r}_{\text{screen}}} \int d\mathbf{r} f(\mathbf{r}) e^{-i\mathbf{q} \cdot \mathbf{r}} = AS e^{i\mathbf{k}_{\text{out}} \cdot \mathbf{r}_{\text{screen}}} F(\mathbf{q})$$

where $\mathbf{q} = \mathbf{k}_{\text{out}} - \mathbf{k}_{\text{in}}$. The measured intensity of the reflection will be square of this amplitude

$$A^2 S^2 |F(\mathbf{q})|^2$$

Friedel and Bijvoet mates

For every reflection corresponding to a point \mathbf{q} in the reciprocal space, there is another reflection of the same intensity at the opposite point $-\mathbf{q}$. This opposite reflection is known as the *Friedel mate* of the original reflection. This symmetry results from the mathematical fact that the density of electrons $f(\mathbf{r})$ at a position \mathbf{r} is always a real number. As noted above, $f(\mathbf{r})$ is the inverse transform of its Fourier transform $F(\mathbf{q})$; however, such an inverse transform is a complex number in general. To ensure that $f(\mathbf{r})$ is real, the Fourier transform $F(\mathbf{q})$ must be such that the Friedel mates $F(-\mathbf{q})$ and $F(\mathbf{q})$ are complex conjugates of one another. Thus, $F(-\mathbf{q})$ has the same magnitude as $F(\mathbf{q})$ —that is, $|F|(\mathbf{q}) = |F|(-\mathbf{q})$ —but they have the opposite phase, i.e., $\varphi(\mathbf{q}) = -\varphi(-\mathbf{q})$

$$F(-\mathbf{q}) = |F(-\mathbf{q})| e^{i\phi(-\mathbf{q})} = F^*(\mathbf{q}) = |F(\mathbf{q})| e^{-i\phi(\mathbf{q})}$$

The equality of their magnitudes ensures that the Friedel mates have the same intensity $|F|^2$. This symmetry allows one to measure the full Fourier transform from only half the reciprocal space, e.g., by rotating the crystal slightly more than a 180° , instead of a full turn. In crystals with significant symmetry, even more reflections may have the same intensity (Bijvoet mates); in such cases, even less of the reciprocal space may need to be measured, e.g., slightly more than 90° .

The Friedel-mate constraint can be derived from the definition of the inverse Fourier transform

$$f(\mathbf{r}) = \int \frac{d\mathbf{q}}{(2\pi)^3} F(\mathbf{q}) e^{i\mathbf{q} \cdot \mathbf{r}} = \int \frac{d\mathbf{q}}{(2\pi)^3} |F(\mathbf{q})| e^{i\phi(\mathbf{q})} e^{i\mathbf{q} \cdot \mathbf{r}}$$

Since Euler's formula states that $e^{ix} = \cos(x) + i \sin(x)$, the inverse Fourier transform can be separated into a sum of a purely real part and a purely imaginary part

$$f(\mathbf{r}) = \int \frac{d\mathbf{q}}{(2\pi)^3} |F(\mathbf{q})| e^{i(\phi + \mathbf{q} \cdot \mathbf{r})} = \int \frac{d\mathbf{q}}{(2\pi)^3} |F(\mathbf{q})| \cos(\phi + \mathbf{q} \cdot \mathbf{r}) + i \int \frac{d\mathbf{q}}{(2\pi)^3} |F(\mathbf{q})| \sin(\phi + \mathbf{q} \cdot \mathbf{r}) = I_{\cos}$$

The function $f(\mathbf{r})$ is real if and only if the second integral I_{\sin} is zero for all values of \mathbf{r} . In turn, this is true if and only if the above constraint is satisfied

$$I_{\sin} = \int \frac{d\mathbf{q}}{(2\pi)^3} |F(\mathbf{q})| \sin(\phi + \mathbf{q} \cdot \mathbf{r}) = \int \frac{d\mathbf{q}}{(2\pi)^3} |F(-\mathbf{q})| \sin(-\phi - \mathbf{q} \cdot \mathbf{r}) = -I_{\sin}$$

since $I_{\sin} = -I_{\sin}$ implies that $I_{\sin} = 0$.

Ewald's sphere

Each X-ray diffraction image represents only a slice, a spherical slice of reciprocal space, as may be seen by the Ewald sphere construction. Both \mathbf{k}_{out} and \mathbf{k}_{in} have the same length, due to the elastic scattering, since the wavelength has not changed. Therefore, they may be represented as two radial vectors in a sphere in reciprocal space, which shows the values of \mathbf{q} that are sampled in a given diffraction image. Since there is a slight spread in the incoming wavelengths of the incoming X-ray beam, the values of $|F(\mathbf{q})|$ can be measured only for \mathbf{q} vectors located between the two spheres corresponding to those radii. Therefore, to obtain a full set of Fourier transform data, it is necessary to rotate the crystal through

slightly more than 180° , or sometimes less if sufficient symmetry is present. A full 360° rotation is not needed because of a symmetry intrinsic to the Fourier transforms of real functions (such as the electron density), but "slightly more" than 180° is needed to cover all of reciprocal space within a given resolution because of the curvature of the Ewald sphere (*add Figure to illustrate this*). In practice, the crystal is rocked by a small amount (0.25 - 1°) to incorporate reflections near the boundaries of the spherical Ewald shells.

Patterson function

A well-known result of Fourier transforms is the autocorrelation theorem, which states that the autocorrelation $c(\mathbf{r})$ of a function $f(\mathbf{r})$

$$c(\mathbf{r}) = \int d\mathbf{x} f(\mathbf{x}) f(\mathbf{x} + \mathbf{r}) = \int \frac{d\mathbf{q}}{(2\pi)^3} C(\mathbf{q}) e^{i\mathbf{q}\cdot\mathbf{r}}$$

has a Fourier transform $C(\mathbf{q})$ that is the squared magnitude of $F(\mathbf{q})$

$$C(\mathbf{q}) = |F(\mathbf{q})|^2$$

Therefore, the autocorrelation function $c(\mathbf{r})$ of the electron density (also known as the *Patterson function*^[71]) can be computed directly from the reflection intensities, without computing the phases. In principle, this could be used to determine the crystal structure directly; however, it is difficult to realize in practice. The autocorrelation function corresponds to the distribution of vectors between atoms in the crystal; thus, a crystal of N atoms in its unit cell may have $N(N-1)$ peaks in its Patterson function. Given the inevitable errors in measuring the intensities, and the mathematical difficulties of reconstructing atomic positions from the interatomic vectors, this technique is rarely used to solve structures, except for the simplest crystals.

Advantages of a crystal

In principle, an atomic structure could be determined from applying X-ray scattering to non-crystalline samples, even to a single molecule. However, crystals offer a much stronger signal due to their periodicity.

A crystalline sample is by definition periodic; a crystal is composed of many unit cells repeated indefinitely in three independent directions. Such periodic systems have a Fourier transform that is concentrated at periodically repeating points in reciprocal space known as *Bragg peaks*; the Bragg peaks correspond to the reflection spots observed in the diffraction image. Since the amplitude at these reflections grows linearly with the number N of scatterers, the observed *intensity* of these spots should grow quadratically, like N^2 . In other words, using a crystal concentrates the weak scattering of the individual unit cells into a much more powerful, coherent reflection that can be observed above the noise. This is an example of constructive interference.


In a non-crystalline sample, molecules within that sample would be in random orientations and therefore would have a continuous Fourier spectrum that spreads its amplitude more uniformly and with a much reduced intensity, as is observed in SAXS. More importantly, the orientational information is lost. In the crystal, the molecules adopt the same orientation within the crystal, whereas in a liquid, powder or amorphous state, the observed signal is averaged over the possible orientations of the molecules. Although theoretically possible with sufficiently low-noise data, it is generally difficult to obtain atomic-resolution structures of complicated, asymmetric molecules from such rotationally averaged

scattering data. An intermediate case is fiber diffraction in which the subunits are arranged periodically in at least one dimension.

See also

- Bragg diffraction
- Bravais lattice
- Crystallographic database
- Crystallographic point groups
- Difference density map
- Electron diffraction
- Neutron diffraction
- Powder diffraction
- Scherrer Equation
- Small angle X-ray scattering (SAXS)
- Structure determination
- Wide angle X-ray scattering (WAXS)
- Ptychography

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External links

Tutorials

- Simple, non technical introduction (<http://stein.bioch.dundee.ac.uk/~charlie/index.php?section=1>)
- "Small Molecule Crystalization" (<http://acaschool.iit.edu/lectures04/JLiangXtal.pdf>) (PDF) at Illinois Institute of Technology website
- International Union of Crystallography (<http://iucr.org>)
- Crystallography 101 (<http://www.ruppweb.org/Xray/101index.html>)
- Interactive structure factor tutorial (<http://www.ysbl.york.ac.uk/~cowtan/sfapplet/sfintro.html>), demonstrating properties of the diffraction pattern of a 2D crystal.
- Picturebook of Fourier Transforms (<http://www.ysbl.york.ac.uk/~cowtan/fourier/fourier.html>), illustrating the relationship between crystal and diffraction pattern in 2D.
- Lecture notes on X-ray crystallography and structure determination (http://www.chem.uwec.edu/Chem406_F06/Pages/lectnotes.html#lecture7)

Primary databases

- Protein Data Bank (<http://www.rcsb.org/pdb/home/home.do>) (PDB)
- Nucleic Acid Databank (<http://ndbserver.rutgers.edu/>) (NDB)
- Cambridge Structural Database (<http://www.ccdc.cam.ac.uk/products/csd/>) (CSD)
- Inorganic Crystal Structure Database (<http://www.fiz-karlsruhe.de/icsd.html>) (ICSD)
- Biological Macromolecule Crystallization Database (<http://xpdb.nist.gov:8060/BMCD4/>) (BMCD)

Derivative databases

- PDBsum (<http://www.ebi.ac.uk/thornton-srv/databases/pdbsum/>)
- Proteopedia - the collaborative, 3D encyclopedia of proteins and other molecules (<http://www.proteopedia.org>)
- RNABase (<http://www.rnabase.org/>)
- HIC-Up database of PDB ligands (<http://xray.bmc.uu.se/hicup/>)
- Structural Classification of Proteins database
- CATH Protein Structure Classification
- List of transmembrane proteins with known 3D structure (http://blanco.biomol.uci.edu/Membrane_Proteins_xtal.html)
- Orientations of Proteins in Membranes database

Structural validation

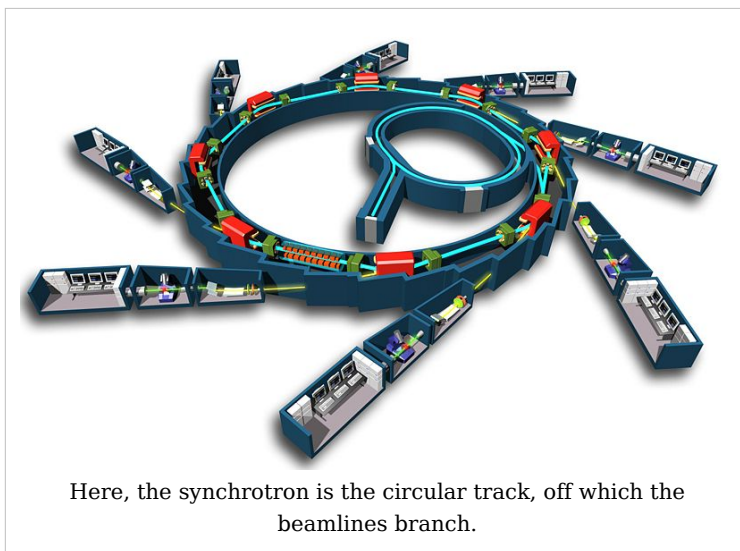
- WHAT-IF structural validation suite (<http://swift.cmbi.kun.nl/WIWWWI/>)
 - Biotech structural validation suite (<http://biotech.ebi.ac.uk/>) (formerly ProCheck)
 - MolProbity structural validation suite (<http://molprobity.biochem.duke.edu/>)
 - ProSA-web (<https://prosa.services.came.sbg.ac.at/prosa.php>)
 - NQ-Flipper (<https://flipper.services.came.sbg.ac.at/>) (check for unfavorable rotamers of Asn and Gln residues)
 - DALI server (<http://www.ebi.ac.uk/dali/>) (identifies proteins similar to a given protein)
-

External links

Neutron Scattering

Synchrotron

A **synchrotron** is a particular type of cyclic particle accelerator in which the magnetic field (to turn the particles so they circulate) and the electric field (to accelerate the particles) are carefully synchronized with the travelling particle beam. The proton synchrotron was originally conceived by Sir Marcus Oliphant^[1]. The honour of the first to publish the idea belongs to Vladimir Veksler, and the first electron synchrotron was constructed by Oliphant's supervisor Edwin McMillan.



Characteristics

While a cyclotron uses a constant magnetic field and a constant-frequency applied electric field (one of these is varied in the synchrocyclotron), both of these fields are varied in the synchrotron. By increasing these parameters appropriately as the particles gain energy, their path can be held constant as they are accelerated. This allows the vacuum chamber for the particles to be a large thin torus. In reality it is easier to use some straight sections between the bending magnets and some bent sections within the magnets giving the torus the shape of a round-cornered polygon. A path of large effective radius may thus be constructed using simple straight and curved pipe segments, unlike the disc-shaped chamber of the cyclotron type devices. The shape also allows and requires the use of multiple magnets to bend the particle beams. Straight sections are required at spacings around a ring for both radiofrequency cavities, and in third generation light sources allow space for insertion devices such as wigglers and undulators.

The maximum energy that a cyclic accelerator can impart is typically limited by the strength of the magnetic field(s) and the minimum radius (maximum curvature) of the particle path.

In a cyclotron the maximum radius is quite limited as the particles start at the center and spiral outward, thus the entire path must be a self-supporting disc-shaped evacuated chamber. Since the radius is limited, the power of the machine becomes limited by the strength of the magnetic field. In the case of an ordinary electromagnet the

field strength is limited by the saturation of the core (when all magnetic domains are aligned the field may not be further increased to any practical extent). The arrangement of the single pair of magnets the full width of the device also limits the economic size of the device.



The interior of the Australian Synchrotron facility. Dominating the image is the storage ring, showing the optical diagnostic beamline at front right. In the middle of the storage ring is the booster synchrotron and linac

Synchrotrons overcome these limitations, using a narrow beam pipe which can be surrounded by much smaller and more tightly focusing magnets. The ability of this device to accelerate particles is limited by the fact that the particles must be charged to be accelerated at all, but charged particles under acceleration emit photons (light), thereby losing energy. The limiting beam energy is reached when the energy lost to the lateral acceleration required to maintain the beam path in a circle equals the energy added each cycle. More powerful accelerators are built by using large radius paths and by using more numerous and more powerful microwave cavities to accelerate the particle beam between corners. Lighter particles (such as electrons) lose a larger fraction of their energy when turning. Practically speaking, the energy of electron/positron accelerators is limited by this radiation loss, while it does not play a significant role in the dynamics of proton or ion accelerators. The energy of those is limited strictly by the strength of magnets and by the cost.

Design and operation

Particles are injected into the main ring at substantial energies by either a linear accelerator or by an intermediate synchrotron which is in turn fed by a linear accelerator. The "linac" is in turn fed by particles accelerated to intermediate energy by a simple high voltage power supply, typically a Cockcroft-Walton generator.

Starting from an appropriate initial value determined by the injection velocity the magnetic field is then increased. The particles pass through an electrostatic accelerator driven by a high alternating voltage. At particle speeds not close to the speed of light the frequency of the accelerating voltage can be made roughly proportional to the current in the bending magnets. A finer control of the frequency is performed by a servo loop which responds to the detection of the passing of the traveling group of particles. At particle speeds approaching light speed the frequency becomes more nearly constant, while the current in the bending magnets continues to increase. The maximum energy that can be applied to the particles (for a given ring size and magnet count) is determined by the saturation of the cores of the bending magnets (the point at which increasing current does not produce

additional magnetic field). One way to obtain additional power is to make the torus larger and add additional bending magnets. This allows the amount of particle redirection at saturation to be less and so the particles can be more energetic. Another means of obtaining higher power is to use superconducting magnets, these not being limited by core saturation.

Large synchrotrons

One of the early large synchrotrons, now retired, is the Bevatron, constructed in 1950 at the Lawrence Berkeley Laboratory. The name of this proton accelerator comes from its power, in the range of 6.3 GeV (then called BeV for billion electron volts; the name predates the adoption of the SI prefix giga-). A number of heavy elements, unseen in the natural world, were first created with this machine. This site is also the location of one of the first large bubble chambers used to examine the results of the atomic collisions produced here.



Modern industrial-scale synchrotrons can be very large (here, Soleil near Paris)

Another early large synchrotron is the Cosmotron built at Brookhaven National Laboratory which reached 3.3 GeV in 1953.^[2]

Until August 2008, the highest energy synchrotron in the world was the Tevatron, at the Fermi National Accelerator Laboratory, in the United States. It accelerates protons and antiprotons to slightly less than 1 TeV of kinetic energy and collides them together. The Large Hadron Collider (LHC), which has been built at the European Laboratory for High Energy Physics (CERN), has roughly seven times this energy. It is housed in the 27 km tunnel which formerly housed the Large Electron Positron (LEP) collider, so it will maintain the claim as the largest scientific device ever built. The LHC will also accelerate heavy ions (such as lead) up to an energy of 1.15 PeV.

The largest device of this type seriously proposed was the Superconducting Super Collider (SSC), which was to be built in the United States. This design, like others, used superconducting magnets which allow more intense magnetic fields to be created without the limitations of core saturation. While construction was begun, the project was cancelled in 1994, citing excessive budget overruns — this was due to naïve cost estimation and economic management issues rather than any basic engineering flaws. It can also be argued that the end of the Cold War resulted in a change of scientific funding priorities that contributed to its ultimate cancellation.

While there is still potential for yet more powerful proton and heavy particle cyclic accelerators, it appears that the next step up in electron beam energy must avoid losses due to synchrotron radiation. This will require a return to the linear accelerator, but with devices significantly longer than those currently in use. There is at present a major effort to

design and build the International Linear Collider (ILC), which will consist of two opposing linear accelerators, one for electrons and one for positrons. These will collide at a total center of mass energy of 0.5 TeV.

However, synchrotron radiation also has a wide range of applications (see synchrotron light) and many 2nd and 3rd generation synchrotrons have been built especially to harness it. The largest of those 3rd generation synchrotron light sources are the European Synchrotron Radiation Facility (ESRF) in Grenoble, France, the Advanced Photon Source (APS) near Chicago, USA, and SPring-8 in Japan, accelerating electrons up to 6, 7 and 8 GeV, respectively.

Synchrotrons which are useful for cutting edge research are large machines, costing tens or hundreds of millions of dollars to construct, and each beamline (there may be 20 to 50 at a large synchrotron) costs another two or three million dollars on average. These installations are mostly built by the science funding agencies of governments of developed countries, or by collaborations between several countries in a region, and operated as infrastructure facilities available to scientists from universities and research organisations throughout the country, region, or world. More compact models, however, have been developed, such as the Compact Light Source.

List of installations

Synchrotron	Location & Country	Energy (GeV)	Circumference (m)	Commissioned	Decommissioned
Advanced Photon Source (APS)	Argonne National Laboratory, USA	7.0	1104	1995	
ISIS	Rutherford Appleton Laboratory, UK	0.8	163	1985	
Australian Synchrotron	Melbourne, Australia	3	216	2006	
LNLS	Campinas, Brazil	1.37	93.2	1997	
SESAME	Allaan, Jordan	2.5	125	Under Design	
Bevatron	Lawrence Berkeley Laboratory, USA	6	114	1954	1993
Advanced Light Source	Lawrence Berkeley Laboratory, USA	1.9	196.8	1993	
Cosmotron	Brookhaven National Laboratory, USA	3	72	1953	1968
Nimrod	Rutherford Appleton Laboratory, UK	7		1957	1978
Alternating Gradient Synchrotron (AGS)	Brookhaven National Laboratory, USA	33	800	1960	
Stanford Synchrotron Radiation Lightsource	SLAC National Accelerator Laboratory, USA	3	234	1973	
Cornell High Energy Synchrotron Source (CHESS)	Cornell University, USA	5.5	768	1979	

Soleil	Paris, France	3	354	2006	
Shanghai Synchrotron Radiation Facility (SSRF)	Shanghai, China	3.5	432	2007	
Proton Synchrotron	CERN, Switzerland	28	628.3	1959	
Tevatron	Fermi National Accelerator Laboratory, USA	1000	6300	1983	
Swiss Light Source	Paul Scherrer Institute, Switzerland	2.8	288	2001	
Large Hadron Collider (LHC)	CERN, Switzerland	7000	26659	2008	
BESSY II	Helmholtz-Zentrum Berlin in Berlin, Germany	1.7	240	1998	
European Synchrotron Radiation Facility (ESRF)	Grenoble, France	6	844	1988	
MAX-I	MAX-lab, Sweden	0.55	30	1986	
MAX-II	MAX-lab, Sweden	1.5	90	1997	
MAX-III	MAX-lab, Sweden	0.7	36	2008	
ELETTRA	Trieste, Italy	2-2.4	260	1993	
Diamond Light Source	Oxfordshire, UK	3	561.6	2002	
DORIS III	DESY, Germany	4.5	289	1980	
PETRA II	DESY, Germany	12	2304	1995	2007
Canadian Light Source	University of Saskatchewan, Canada	2.9	171	2002	
SPring-8	RIKEN, Japan	8	1436	1997	
Taiwanese National Synchrotron Radiation Research Center	Hsinchu Science Park, Taiwan	3.3	518.4	2008	
Synchrotron Light Research Institute (SLRI)	Nakhon Ratchasima, Thailand	1.2	81.4	2004	
Indus 1	Raja Ramanna Centre for Advanced Technology, Indore, India	0.45		1999	
Indus 2	Raja Ramanna Centre for Advanced Technology, Indore, India	2.5	36	2005	
Synchrophasotron	JINR, Dubna, USSR	10	180	1957	2005
U-70	IHEP, Protvino, USSR	70		1967	

CAMD	LSU, Louisiana, US	1.5	-	-	
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- Note: in the case of colliders, the quoted power is often double what is shown here. The above table shows the power of one beam but if two opposing beams collide head on, the effective power is doubled.

Applications

- Life sciences: protein and large molecule crystallography
- Drug discovery and research
- "Burning" computer chip designs into metal wafers
- Studying molecule shapes and protein crystals
- Analyzing chemicals to determine their composition
- Observing the reaction of living cells to drugs
- Inorganic material crystallography and microanalysis
- Fluorescence studies
- Semiconductor material analysis and structural studies
- Geological material analysis
- Medical imaging
- Proton therapy to treat some forms of cancer

See also

- List of synchrotron radiation facilities
- Synchrotron X-ray tomographic microscopy
- Energy amplifier
- Superconducting Radio Frequency

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- [1] Nature 407, 468 (28 September 2000) (<http://www.nature.com/nature/journal/v407/n6803/full/407468a0.html>).
- [2] The Cosmotron (<http://www.bnl.gov/bnlweb/history/cosmotron.asp>)

External links

- Australian Synchrotron (<http://www.synchrotron.org.au>)
- Diamond UK Synchrotron (<http://www.diamond.ac.uk>)
- Lightsources.org (<http://www.lightsources.org/cms/>)
- CERN Large Hadron Collider (<http://lhc-new-homepage.web.cern.ch/lhc-new-homepage>)
- Synchrotron Light Sources of the World (http://www-als.lbl.gov/als/synchrotron_sources.html)
- A Miniature Synchrotron: (<http://www.technologyreview.com/Biotech/20149/>) room-size synchrotron offers scientists a new way to perform high-quality x-ray experiments in their own labs, *Technology Review*, February 04, 2008
- Brazilian Synchrotron Light Laboratory (http://www.lnls.br/lnls/cgi/cgilua.exe/sys/start.htm?UserActiveTemplate=lnls_2007_english&tpl=home)
- Podcast interview (<http://omegataupodcast.net/2009/03/28/11-synchrotron-radiation-science-at-esrf/>) with a scientist at the European Synchrotron

Radiation Facility

See also

Neutron diffraction

Neutron diffraction is a method for the determination of the atomic and/or magnetic structure of a material. It can be equally well applied to study crystalline solids (see crystallography), gasses, liquids or amorphous materials. Neutron diffraction is a form of elastic scattering where the neutrons exiting the experiment have more or less the same energy as the incident neutrons. The technique is similar to X-ray diffraction but the different type of radiation gives complementary information. A sample to be examined is placed in a beam of thermal or cold neutrons and the intensity pattern around the sample gives information of the structure of the material.

Description

Principle

Neutrons are particles found in the atomic nucleus of almost all atoms, but they are bound. The technique requires free neutrons and these normally do not occur in nature, because they have limited life-time. In a nuclear reactor, however, neutrons can be set free through nuclei decay particularly when fission occurs. All quantum particles can exhibit wave phenomena we typically associate with light or sound. Diffraction is one of these phenomena; it occurs when waves encounter obstacles whose size is comparable with the wavelength. If the wavelength of a quantum particle is short enough, atoms or their nuclei can serve as diffraction obstacles. When a beam of neutrons emanating from a reactor is slowed down and selected properly by their speed, their wavelength lies near one Ångström (0.1 nanometer), the typical separation between atoms in a solid material. Such a beam can then be used to perform a diffraction experiment. Impinging on a crystalline sample it will scatter under a limited number of well-defined angles according to the same Bragg's law that describes X-ray diffraction.

Instrumental requirements

A neutron diffraction measurement requires a neutron source (e.g. a nuclear reactor or spallation source), a sample (the material to be studied), and a detector. Samples sizes are large compared to those used in X-ray diffraction. The technique is therefore mostly performed as powder diffraction. At a research reactor other components such as crystal monochromators or filters may be needed to select the desired neutron wavelength. Some parts of the setup may also be movable. At a spallation source the time of flight technique is used to sort the energies of the incident neutrons, so no monochromator is needed, just a bunch of electronics. (Higher energy neutrons are faster - v. simple)

Nuclear scattering

Neutrons interact with matter differently than x-rays. X-rays interact primarily with the electron cloud surrounding each atom. The contribution to the diffracted x-ray intensity is therefore larger for atoms with a large atomic number (Z) than it is for atoms with a small Z . On the other hand, neutrons interact directly with the *nucleus* of the atom, and the contribution to the diffracted intensity is different for each isotope; for example, regular hydrogen and deuterium contribute differently. It is also often the case that light (low Z) atoms contribute strongly to the diffracted intensity even in the presence of large Z atoms. The scattering length varies from isotope to isotope rather than linearly with the atomic number. An element like Vanadium is a strong scatterer of X-rays, but its nuclei hardly scatter neutrons, which is why it often used as a container material. Non-magnetic neutron diffraction is directly sensitive to the positions of the nuclei of the atoms.

A major difference with X-rays is that the scattering is mostly due to the tiny nuclei of the atoms. That means that there is no need for an atomic form factor to describe the shape of the electron cloud of the atom and the scattering power of an atom does not fall off with the scattering angle as it does for X-rays. Diffractograms therefore can show strong well defined diffraction peaks even at high angles, particularly if the experiment is done at low temperatures. Many neutron sources are equipped with liquid helium cooling systems that allow to collect data at temperatures down to 4.2 K. The superb high angle (i.e. high *resolution*) information means that the data can give very precise values for the atomic positions in the structure. On the other hand, Fourier maps (and to a lesser extent difference Fourier maps) derived from neutron data suffer from series termination errors, sometimes so much that the results are meaningless.

Magnetic scattering

Although neutrons are uncharged, they carry a spin, and therefore interact with magnetic moments, including those arising from the electron cloud around an atom. Neutron diffraction can therefore reveal the microscopic magnetic structure of a material^[1].

Magnetic scattering does require an atomic form factor as it is caused by the much larger electron cloud around the tiny nucleus. The intensity of the magnetic contribution to the diffraction peaks will therefore dwindle towards higher angles.

History

The first neutron diffraction experiments were carried out in 1945 by Ernest O. Wollan using the Graphite Reactor at Oak Ridge. He was joined shortly thereafter (June 1946)^[2] by Clifford Shull, and together they established the basic principles of the technique, and applied it successfully to many different materials, addressing problems like the structure of ice and the microscopic arrangements of magnetic moments in materials. For this achievement Shull was awarded one half of the 1994 Nobel Prize in Physics. Wollan had died in the 1990s. (The other half of the 1994 Nobel Prize for Physics went to Bert Brockhouse for development of the inelastic scattering technique at the Chalk River facility of AECL. This also involved the invention of the triple axis spectrometer). Brockhouse and Shull jointly take the somewhat dubious distinction of the longest gap between the work being done (1946) and the Nobel Prize being awarded (1994).

Uses

Neutron diffraction is closely related to X-ray powder diffraction^[3]. In fact the single crystal version of the technique is less commonly used because currently available neutron sources require relatively large samples and large single crystals are hard or impossible to come by for most materials. Future developments, however, may well change this picture. Because the data is typically a 1D powder diffractogram they are usually processed using Rietveld refinement. In fact the latter found its origin in neutron diffraction (at Petten in the Netherlands) and was later extended for use in X-ray diffraction.

One practical application of elastic neutron scattering/diffraction is that the lattice constant of metals and other crystalline materials can be very accurately measured. Together with an accurately aligned micropositioner a map of the lattice constant through the metal can be derived. This can easily be converted to the stress field experienced by the material. This has been used to analyse stresses in aerospace and automotive components to give just two examples. This technique has led to the development of dedicated stress diffractometers, such as the ENGIN-X instrument at the ISIS neutron source.

Hydrogen, null-scattering and contrast variation

Neutron diffraction can be used to establish the structure of low atomic number materials like proteins and surfactants much more easily with lower flux than at a synchrotron radiation source. This is because some low atomic number materials have a higher cross section for neutron interaction than higher atomic weight materials.

One major advantage of neutron diffraction over X-ray diffraction is that the latter is rather insensitive to the presence of hydrogen in a structure, whereas the nuclei ^1H and $^2\text{H}=\text{D}$ are strong scatterers for neutrons. This means that the position of hydrogen in a crystal structure and its thermal motions can be determined far more precisely with neutrons. In addition the scattering lengths (structure factors in x-ray parlance) of H and D have opposite sign, which allows contrast variation. In fact there is a particular isotope ratio for which the contribution of the element would cancel, this is called null-scattering. In practice however it is not desirable to work with the relatively high concentration of H in such a sample. The scatter by H-nuclei has a large inelastic component and this creates a large continuous background that is more or less independent of scattering angle. The elastic pattern typically consists of sharp Bragg reflections if the sample is crystalline. They tend to drown in the inelastic background. This is even more serious when the technique is used for the study of liquid structure. Nevertheless, by preparing samples with different isotope ratios it is possible to vary the scattering contrast enough to highlight one element in an otherwise complicated structure. The variation of other elements is possible but usually rather expensive. Hydrogen is inexpensive and particularly interesting because it plays an exceptionally large role in biochemical structures and is difficult to study structurally in other ways.

Further reading

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See also

- Bragg diffraction
- Crystallography
- Crystallographic database
- Diffraction
- Electron crystallography
- Electron diffraction
- Neutron crystallography
- Neutron Diffraction at OPAL
- X-ray crystallography
- X-ray diffraction
- X-ray scattering techniques

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- [2] Clifford Shull: Early development of neutron scattering. Rev. Mod. Phys. 67 (1995) 753–757
- [3] *Neutron powder diffraction* by Richard M. Ibberson and William I.F. David, Chapter 5 of Structure determination from powder diffraction data IUCr monographs on crystallography, Oxford scientific publications 2002, ISBN 0-19-850091-2

External links

- Institute of Physics Neutron Scattering Group (http://www.iop.org/Our_Activities/Groups_and_Divisions/Subject_Groups/Neutron_Scattering/page_5477.html) (accessed April 2007)
- German Committee Research with Neutrons (<http://www.physik.uni-kiel.de/kfn/index-engl.php>)
- Engineering instrument at ISIS (<http://www.isis.rl.ac.uk/Engineering/>)
- National Institute of Standards and Technology Center for Neutron Research (<http://www.ncnr.nist.gov/>)

Neutron time-of-flight scattering

In **Neutron time-of-flight scattering**, a form of inelastic neutron scattering, the initial position and velocity of a pulse of neutrons is fixed, and their final position and the time after the pulse that the neutrons are detected are measured. By the principle of conservation of momentum, these pairs of coordinates may be transformed into momenta and energies for the neutrons, and the experimentalist may use this information to calculate the momentum and energy transferred to the sample. Inverse geometry spectrometers are also possible. In this case, the final position and velocity are fixed, and the incident coordinates varied.

Time-of-flight scattering can be performed at either a research reactor or a spallation source.

External links

Existing time-of-flight spectrometers at spallation sources:

- LRMECS ^[1] and HRMECS ^[2] at Argonne National Laboratory
- Pharos ^[3] at LANSCE ^[4], s Lujan Center ^[4] at Los Alamos National Laboratory
- MAPS ^[5], MARI ^[6], HET ^[7] at the ISIS neutron source

Soon to be time-of-flight spectrometers at spallation sources:

- ARCS ^[8], CNCS ^[9], and SEQUOIA ^[10] at the Spallation Neutron Source
- MERLIN ^[11] and LET ^[12] at the ISIS neutron source

Existing time-of-flight spectrometers at research reactors:

- DCS ^[13] and FCS ^[14] at the NIST laboratories in Maryland.
- IN4 ^[15], IN5 ^[16], and IN6 ^[17] at the Institut Laue-Langevin
- TOFTOF ^[18] at the Forschungsneutronenquelle Heinz Maier-Leibnitz

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- [1] <http://www.pns.anl.gov/instruments/lrmecs/>
 - [2] <http://www.pns.anl.gov/instruments/hrmecs/>
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 - [5] <http://www.isis.rl.ac.uk/excitations/maps>
 - [6] <http://www.isis.rl.ac.uk/excitations/mari>
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 - [13] <http://www.ncnr.nist.gov/instruments/dcs/>
 - [14] <http://www.ncnr.nist.gov/instruments/fcs>
 - [15] <http://www.ill.fr/YellowBook/IN4/>
 - [16] <http://www.ill.fr/YellowBook/IN5/>
 - [17] <http://www.ill.fr/YellowBook/IN6/>
 - [18] <http://www.frm2.tum.de/toftof/>
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Neutron scattering facilities

Australia: ANSTO - HIFAR Reactor (Sydney)

Canada: Chalk River Laboratories Canadian Neutron Facility

England: ISIS Spallation Source, Rutherford - Appleton Laboratory (Oxford)

France: ILL - Institut Laue Langevin LLB - Léon Brillouin Lab. at CEA (Saclay)

Germany: FRM II - Technical University (Garching) FRJ-2 at FZ Jülich, member of HGF (Bonn) GKSS - Institute for Materials Research (Hamburg) HMI - Hahn-Meitner Institute (Berlin), member of HGF

Hungary: KFKI Research Institutes (Budapest)

India: Dhruva, CIRUS and Apsara: Bhabha Atomic Research Centre, Mumbai

Japan: JAERI - J. Atomic Energy Research Inst. KENS - High Energy Accelerator Organisation, KEK KURRI - Research Reactor Institute (Kyoto) JSNS - (part of the Japan proton accelerator research complex (J-PARC)

Netherlands: IRI - Interfaculty Reactor Institute , Delft University of Technology

Russia: IBR Fast Pulsed Reactors (Dubna) JINR - Joined Institute for Nuclear Research (Dubna)

Sweden: NFL - Studsvik Neutron Research Laboratory (Studsvik)

Switzerland: PSI - Paul Scherrer Institute

United States: HFBR - High Flux Beam Reactor (Brookhaven) IPNS - Intense Pulsed Neutron Source, ANL (Argonne) LANSCE - Los Alamos Neutron Science Center (Los Alamos) NIST - Center for Neutron Research (Washington) ORNL - High Flux Isotope Reactor (Oak Ridge) SNS - Spallation Source, Oak Ridge (Tennessee)

[The European Spallation Source (ESS)] [www.esss.se; www.esshungary.eu]

External links

Vibrational Circular Dichroism (VCD)

Vibrational modes

Paracrystalline state/Paracrystal

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Summary

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FCS, FCCS, FRET, FRAP

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